

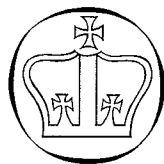
CRYSTAL
ORIENTATION
MANUAL

ELIZABETH A. WOOD

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ORIENTATION
MANUAL

BY ELIZABETH A. WOOD

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Preface

This manual collects in convenient form basic information necessary for the orientation of crystals for purposes of physical measurement and experimentation. By "orient" is meant the process of adjusting the crystal until its orientation relative to some fixed holder is that desired for purposes of taking data or cutting a slice, rod, or other specimen for subsequent experiments. What is not meant is the determination of the existing orientation of a grain, as in the case of studies of preferred orientation in cold worked metals, a subject discussed by C. S. Barrett in *The Structure of Metals* (1952).

The crystal to be oriented may be several inches or a few millimeters in diameter; it may or may not exhibit crystal faces; it may be transparent or opaque.

Many chemists, physicists, engineers, and technicians who are today confronted with the problem of obtaining a slice or rod of suitable orientation for their experiments have not had crystallographic training: it is for these that this manual was written.

A course in crystallography would be excellent preparation for crystal-orienting work. The following texts will provide helpful background: *An Introduction to Crystallography* by F. C. Phillips (1956), the introductory chapters (2, 3, and 4) of *Chemical Crystallography* by C. W. Bunn (1961), *Crystallography* by P. Terpstra and L. W. Codd (1961), and certain chapters (1, 2, 5, and 9) of *The Structure of Metals* by C. S. Barrett (1952). Only the last discusses crystal orienting in the sense in which it is used in this manual.

I would like to express my gratitude to Walter L. Bond, whose physical intuition, ingenuity, and three-dimensional perspicacity have made him outstanding in many aspects of crystal work and who, with great kindness and patience, taught me how to orient crystals many years ago.

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Introduction

This manual has been designed to minimize the amount of reading required to obtain the necessary information. Since readers with previous experience may not need some of the information, it has been supplied in discrete packages which may be referred to or not, according to the needs of the reader.

There is first a step-by-step procedure for orienting crystals. It may not cover all cases, but it will fit many of them. The rest of the sections are information sections to supply the information needed to proceed in accordance with the procedure outlined. A sketch of the crystal showing what is known about it at each stage will help.

Section 24, Examples of Pitfalls, gives four accounts of actual difficulties encountered, and may help you to avoid similar pitfalls.

The precision of orientation required and the nature of the initial specimen and of the final sample to be cut from it will determine the details of the procedure to be followed. In some cases, visual examination coupled with a knowledge of the symmetry of the crystal may suffice. For accurate orientation of the final sample it will nearly always be necessary to find the orientation of known planes of atoms in the specimen.

CAUTION: Exposure to the direct x-ray beam, even for one second, may result in a skin burn which will not heal. Longer exposures to scattered radiation are dangerous. Check all equipment with films in black paper envelopes placed where the operator stands. If the film is noticeably darkened during one week of operation, better radiation protection is needed. A 1/16-in. thick sheet of lead may be used.

1. Procedure for Orienting a Crystal

(Numbers in parentheses refer to information sections (§) which follow.)

A. PRELIMINARY INFORMATION

1. Know the symmetry of the crystal to be oriented. It has the symmetry of one of the 32 point groups. (See §4 and Pitfall 3 of §24.)
2. Sketch the orientation of the plane being sought, relative to the crystallographic axes (§3, §5) and to the symmetry elements (§2, §4).
3. Determine the number of planes which are crystallographically equivalent to the one being sought. It may be more advantageous to use one rather than another (§5).

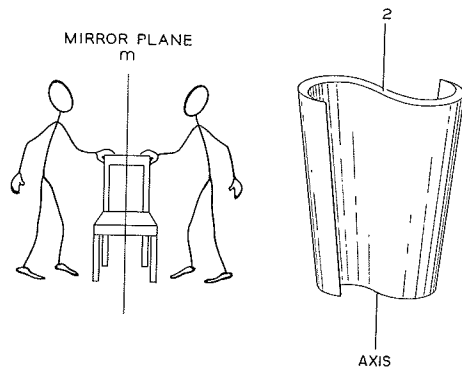
For goniometer work (skip to B if taking a Laue photograph):

4. Look up the lattice constants of the crystal (§6).
5. Use the lattice constants to obtain the d for the plane being sought (§7). Remember that in some cases there are "missing reflections" (§8, §11).
6. Determine the Bragg reflecting angle θ for reflection of the x rays being used from the atomic plane being sought (§8, §9).

B. ORIENTING THE CRYSTAL

1. Before mounting the crystal, think through to its ultimate use and be sure you can transfer it as needed without losing orientation (§14). Make sure that the specimen is in fact a single crystal (§12, Appendices).
2. If any directions are already known (from growth-faces, etch pits, ridges on grown rod, etc.), use these to get a head start (§12). If the crystal is transparent, examination in polarized light may be very useful in determining orientation (Appendix I).
3. If the approximate location of the desired atomic plane is known, mount the crystal (§14, §15), set the Geiger counter (§16) for the chosen reflection (§8, §9, and §10), and look for the maximum Geiger counter response by changing the angle of the goniometer arm. You will not be certain of your plane until it can be rotated 360° around its own normal with a continuous "maximum" reflection (§16).
4. If the approximate location of the plane is not known, take a Laue photograph (§17) and adjust the crystal accordingly (§18). Some people prefer to try the goniometer first, and only take a Laue photograph in case of trouble. This may lead to error, as in pitfalls number 1 and 4 (§24.)
5. When you are close to the desired orientation, "bring it in" with the goniometer (§16).

2. Symmetry



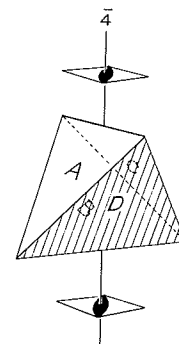
Your right and left hands are related by a mirror plane (also called “symmetry plane”). So are the right and left halves of a chair. If you look at your right hand in a mirror, it looks like your left. Find nine planes of symmetry in a cube.

When an object rotated 360° occupies the same position in space n times, the axis is an n -fold axis. Find in a cube: three 4-fold axes, four 3-fold axes, six 2-fold axes.

Definition of a center of symmetry: A point such that any feature of a crystal encountered a given distance from the point in one direction is also encountered the same distance from the point in the opposite direction.

The $\bar{2}$ -, $\bar{3}$ -, $\bar{4}$ -, and $\bar{6}$ -fold axes are “inversion axes,” a combination of rotation with inversion through a center. Note that this operation differs in result from the operation of a true n -fold axis and a center of symmetry.

Example: A tetrahedron, made of four equilateral triangles with their edges together, has a 4-fold inversion axis as shown ∇ . Face A , rotated clockwise 90° around the axis and then inverted through a center, becomes face D . The tetrahedron does not have either a 4-fold axis or a center of symmetry.

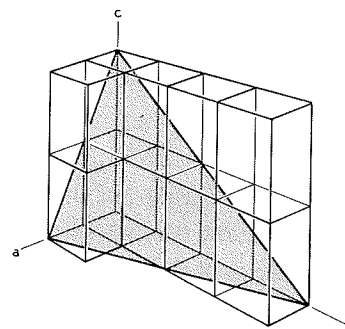


Hermann-Mauguin symbols for the 32 point groups give the symmetry elements of each class (§4). The m stands for mirror plane and the number stands for an axis of rotation. If the plane is normal to the axis, it is placed under the number; if parallel, beside it. Only the necessary symbols are given. Thus, $3m$ describes a 3-fold axis with a plane parallel to it, but, in order to “satisfy” the 3-fold symmetry, there must also be two other planes parallel to the axis, each 120° to the first. Since the “3” indicates this, it is not necessary to mention these planes in the symbol. Since only one plane can be perpendicular to the principal axis, it is obvious that $6/mmm$ should be read “ $6/m\ mm$ ” referring to one plane normal to the 6-fold axis and two parallel to it, each appropriately repeated in accordance with the 6-fold axis.

These axes, planes, etc., of symmetry are known as *symmetry elements*.

3. Crystallographic Axes

It is convenient to describe a solid object in terms of three-dimensional reference axes, describing the crystallographic planes by their intercepts on these axes. Since crystals are composed of very small units of structure ("unit cells"), varying in shape and size from one crystal to another, it is simplest to use the lengths of these cell edges as units of measurement. For example, in the figure, the heavily marked plane is said to intersect the a axis at two units, the c axis at two units, and the b axis at four units. (Because the height of the building blocks is greater than the width, the actual length of the two-unit intercept on c is greater than that of the two-unit intercept on a .) If the building blocks are not rectangular, axes are used which are not perpendicular to each other.



A summary of the relations among the units along various axes and the angles that the axes make with each other in the various crystal "systems" is given below. Here α is the angle between b and c , β is the angle between a and c , and γ is the angle between a and b .

Cubic	$a = b = c$	$\alpha = \beta = \gamma = 90^\circ$
Tetragonal	$a = b \neq c$	$\alpha = \beta = \gamma = 90^\circ$
Orthorhombic ¹	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90^\circ$
Hexagonal	$a = a' = b \neq c$	$\alpha = \beta = 90^\circ, \gamma = 120^\circ$ (See §5.)
[Rhombohedral	$a = b = c$	$\alpha = \beta = \gamma \neq 90^\circ$ (See Appendix III.)]
Monoclinic	$a \neq b \neq c$	$\alpha = \gamma = 90^\circ, \beta > 90^\circ$
Triclinic	$a \neq b \neq c$	$\alpha \neq \beta \neq \gamma$

The unit length of a particular axis is commonly called the length of the axis. Where axes are required by symmetry to be the same length, they may be indicated by the same letter. For example, the three cubic axes may be designated a_1, a_2, a_3 ; the three tetragonal axes may be designated a_1, a_2, c .

¹ "Orthorhombic" was sometimes shortened to "rhombic" in the early literature.

4. The 32 Crystal Classes

The crystallographic point groups are those groups of symmetry elements which can operate on a crystal structure and leave one point unmoved. Only 32 such groups are possible.

Crystals having any particular point-group symmetry are said to belong to a particular "class." Many different cumbersome names and numbering systems have been used for the 32 classes in the past. Increasingly, the point-group designation is being preferred to such names. Relationship to the old nomenclature is easily established via the assemblage of symmetry elements. A table relating the point-group designation to the names used in P. Groth's *Physikalische Kristallographie* (1905) is given on page 137 of *Crystallometry* by P. Terpstra and L. W. Codd (1961), taken from *Mineralogische Tabellen* by H. Strunz (Leipzig, 1941).

On the §4 chart, "The Symmetries of the 32 Point Groups," the diagram on the left in each box is a perspective view of the symmetry elements (§2) of that point group. Planes may be white or shaded. Axes are tipped with the appropriate symbol (see below). Crystallographic axes (§3) are also shown if they differ in orientation from the symmetry axes.

The stereographic projections to the right of the perspective diagrams of the symmetry elements for each point group show the stereographic projection of the symmetry elements and of the "poles" (normals) of the faces of the most general "form" $\{hkl\}$ (§5), plotted as described in §25. Solid dots denote poles falling in the near hemisphere; open circles denote poles falling in the far hemisphere. Symmetry axes are denoted by \dagger (2-fold), \blacktriangle (3-fold), \blacksquare (4-fold), and \bullet (6-fold), open figures denote inversion axes, and a c in the corner denotes a center of symmetry (§2). A symmetry plane is not plotted by its pole, but by the trace of the intersection of the plane with the sphere (§25).

The two symbols, separated by a dash, that are shown beneath each pair of diagrams, are the Schoenflies symbol (first) and the Hermann-Mauguin symbol for the point group. Some discussion of the Hermann-Mauguin symbols is given in §2.

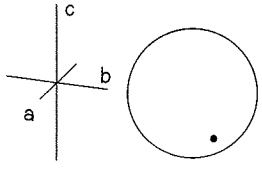
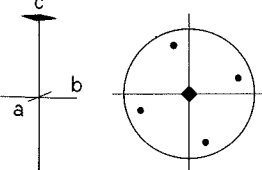

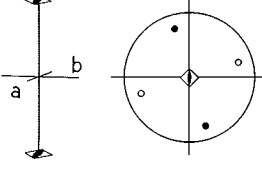
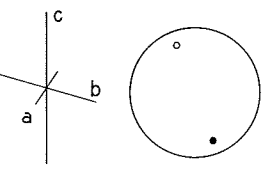
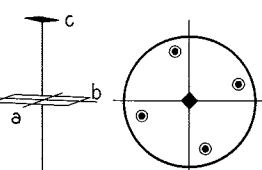
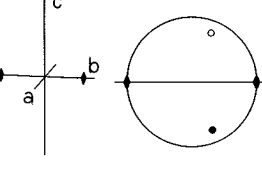
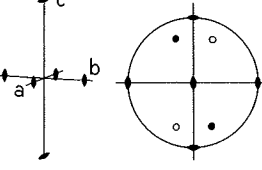
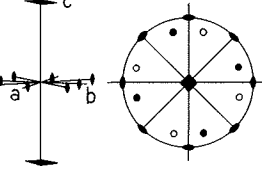
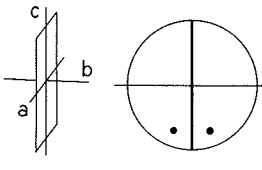
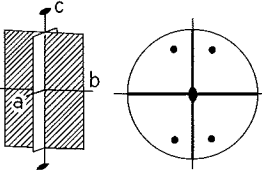
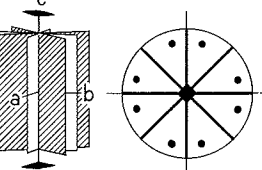


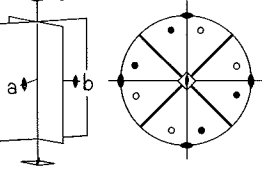
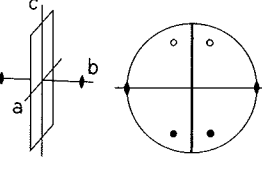
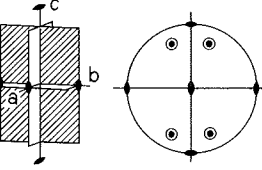
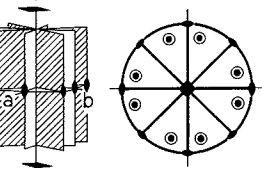
NOTE: In some crystals belonging to point group D_3-32 , the 2-fold symmetry axes are at 90° to the a crystallographic axes (and also, of course, to c). Similarly, in some crystals belonging to point groups $C_{3v} - 3m$ and $D_{3d} - \bar{3}m$, the symmetry planes are parallel to the crystallographic axes rather than at 90° to them, as shown. The space-group symbol for your crystal (§11) will tell you which orientation is applicable. In D_3 , if the 2 is separated from the 3 by a 1, the symmetry axes are at 90° to the a crystallographic axes; if parallel, the 2 is next to the 3 and may or may not be followed by a 1. In C_{3v} , if the m is separated from the 3 by a 1, the symmetry planes are parallel to the crystallographic axes; if they are normal to the a axes, the m is next to the 3 and may or may not be followed by a 1. The same convention is followed for $\bar{3}m$. Thus:

$312, 2 \perp a; 321, 2 \parallel a$

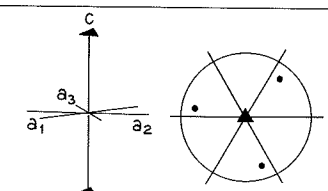
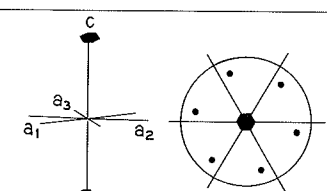
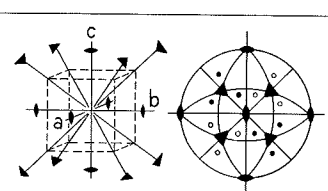
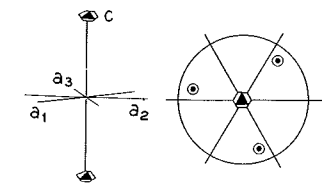
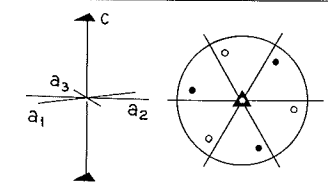
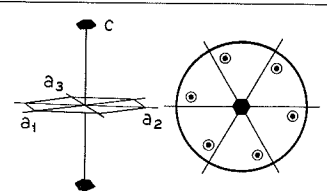
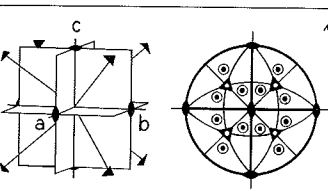
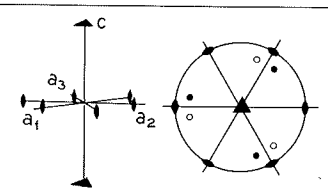
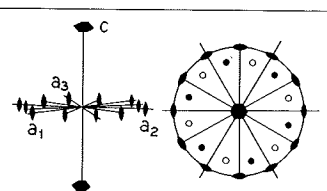
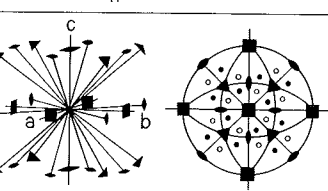
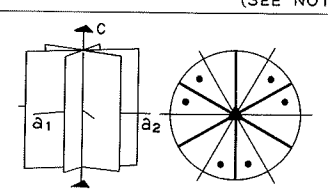
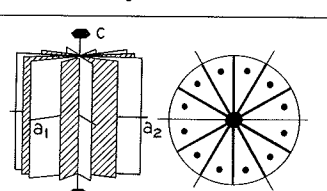
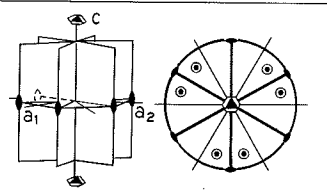
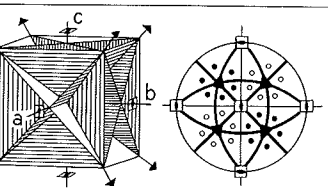
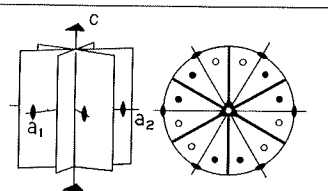
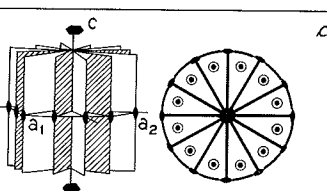
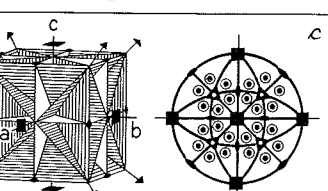
$31m, m \parallel a; 3ml, m \perp a$

$31m, m \parallel a; 3ml, m \perp a$

SYMMETRIES OF THE

TRICLINIC		TETRAGONAL
 $C_1 - 1$		 $C_4 - 4$
 $C_2 - 2$		 $S_4 - \bar{4}$
 $C_i - \bar{1}$		 $C_{4h} - 4/m$
MONOCLINIC	ORTHORHOMBIC	
 $C_2 - 2$	 $D_2 - 222$	 $D_4 - 422$
 $C_s - m$	 $C_{2v} - mm2$	 $C_{4v} - 4mm$
 $C_{2h} - 2/m$	 $D_{2h} - mmm$	 $D_{2d} - \bar{4}2m$
 $C_{2h} - 2/m$	 $D_{2h} - mmm$	 $D_{4h} - 4/mmm$

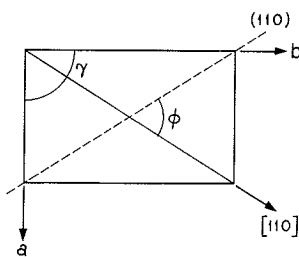
32 POINT GROUPS

TRIGONAL	HEXAGONAL	CUBIC
 <p>$C_3 - 3$</p>	 <p>$C_6 - 6$</p>	 <p>$T - 23$</p>
—	 <p>$C_{3h} - \bar{6}$</p>	—
 <p>$C_{3i} - \bar{3}$</p>	 <p>$C_{6h} - 6/m$</p>	 <p>$T_h - m\bar{3}$</p>
 <p>$D_3 - 32$ (SEE NOTE)</p>	 <p>$D_6 - 622$</p>	 <p>$O - 432$</p>
 <p>$C_{3v} - 3m$ (SEE NOTE)</p>	 <p>$C_{6v} - 6mm$</p>	—
—	 <p>$D_{3h} - \bar{6}m2$</p>	 <p>$T_d - \bar{4}3m$</p>
 <p>$D_{3d} - \bar{3}m$ (SEE NOTE)</p>	 <p>$D_{6h} - 6/mmm$</p>	 <p>$O_h - m\bar{3}m$</p>

5. Miller Indices, "Forms," Multiplicity

Miller indices, hkl , are the reciprocals of the intercepts of a face on the crystal axes. Consider the figure in §3. The intercepts of the plane are 2, 4, 2, naming the axes in the usual order. Since we are only interested in orientation of the plane, the intercepts 1, 2, 1, which are smaller, will describe it just as well. Its Miller indices will be $1/1, 1/2, 1/1$, or $1, 1/2, 1$. Multiplying by 2, to eliminate fractions, we have (212) as the indices of the plane (called "two, one, two"). If the plane had been parallel to c , it would be said to meet c at infinity. Intercepts, 1, 2, ∞ give us Miller indices (210), customarily given in parentheses. Negative Miller indices (bar above number) refer to intercepts at the negative ends of the crystallographic axes.

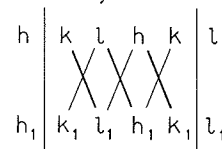
The [210] direction (square brackets) is the direction of a line from the origin to a point whose coordinates are 2, 1, 0. Note that it is only in the case of



planes cutting equal, orthogonal axes (cubic crystals or $hk0$ planes of tetragonal crystals), or planes perpendicular to a single axis and parallel to two others, that the direction $[h_1k_1l_1]$ is normal to the plane $(h_1k_1l_1)$. The figure illustrates this for a (110) plane and [110] direction in an orthorhombic crystal

$$(\gamma = 90^\circ, \phi \neq 90^\circ).$$

The direction $[uvw]$ of the line of intersection of any two planes (hkl) and $(h_1k_1l_1)$ is $u = kl_1 - lk_1$, $v = lh_1 - hl_1$, and $w = hk_1 - kh_1$, conveniently obtained by subtracting the light-diagonal products from the dark-diagonal products in the accompanying diagram. The result $[uvw]$ is the designation of the zone to which these two planes belong. A zone comprises all those planes that are parallel to a given direction. A plane that is common to two zones may be found from the similar relationship $h = vv_1 - ww_1$, $k = ww_1 - uu_1$, $l = uu_1 - vv_1$. If (hkl) is parallel to $[uvw]$, $uh + vk + lw = 0$. The use of these relationships is illustrated in §18.



A form, $\{hkl\}$, curly brackets, is made up of all those faces required by the symmetry, once a given face is mentioned. In order to know how many faces there are in a form ("the multiplicity"), it is of course necessary to know the symmetry of the crystal. E.g., $\{100\}_{m3m} = (100), (010), (001), (\bar{1}00), (0\bar{1}0), (00\bar{1})$.

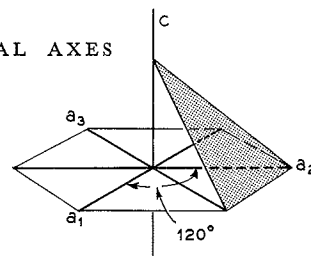
$$\{100\}_{4mm} = (100), (010), (\bar{1}00), (0\bar{1}0).$$

The planes of a form are called "crystallographically equivalent" planes. They have the same physical properties.

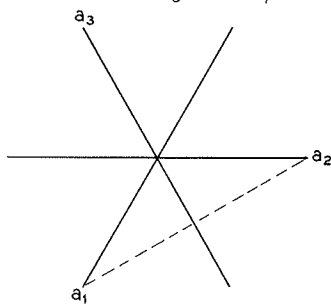
(111) bears a similar relation to [111], referring in class $m3m$ to directions [111], $[\bar{1}\bar{1}\bar{1}]$, $[1\bar{1}\bar{1}]$, $[\bar{1}1\bar{1}]$, $[\bar{1}\bar{1}1]$, $[\bar{1}11]$, $[1\bar{1}1]$, $[1\bar{1}\bar{1}]$. All these directions, being related by symmetry, have the same physical properties.

MILLER-BRAVAIS INDICES FOR HEXAGONAL AXES

In the hexagonal system, there are four crystallographic axes: three horizontal axes of equal length, at 120° to each other, and one vertical axis of a different length from the others. Every plane therefore has four indices, $(hkil)$.

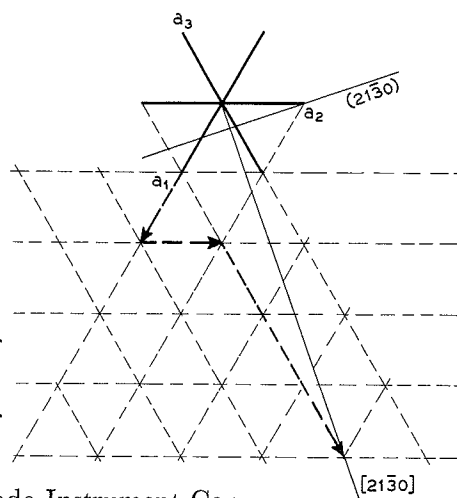


The plane illustrated above cuts a_1 at infinity, a_2 at $+1$, a_3 at -1 , and c at $1/2$. Its indices are therefore $(01\bar{1}2)$ (§5). A plane cutting both a_1 and a_2 at $+1$ will cut a_3 at $-1/2$ and its first three indices will thus be $11\bar{2}$. If it is parallel to c ,



its full symbol will be $(11\bar{2}0)$. Note that in both of these cases the third symbol, i , is the negative sum of the first two, h and k ; i.e., $i = -(h + k)$. Since this is always so, the third symbol is frequently replaced by a dot, as in $(11\cdot0)$ or $(01\cdot2)$.

As in other systems of axes (§5), directions in the hexagonal system are expressed by the smallest integral coordinates of a point on the line from the origin toward the direction in question. Here, because there are four axes, there are four coordinates, and none must be omitted. In the accompanying example, the direction $[21\bar{3}0]$ is found by counting two units along a_1 , one unit along a_2 , -3 units along a_3 , and zero units along c . The trace of the plane $(21\bar{3}0)$ is also shown. Although the direction is normal to the plane with the same indices when the l index is zero, this is not so when l is not zero. Consider the direction that would be obtained if the third index were omitted. It is quite a different direction. Note that the indices of directions are unlike the case of the planes where the intercept on the third axis is obtained as a result of the intercept on the first two.



Wade's isometric lined paper, copyrighted by Wade Instrument Company, Cleveland, Ohio, carries a grid based on three axes at 120° to each other and is thus very useful for work in the hexagonal system. It is available in pads.

A three-digit system of hexagonal direction indices is sometimes used in published papers. For discussion of this and intersystem conversion formulas, see C. S. Barrett, *Structure of Metals* (1952).

6. Sources of Lattice Constant Data

In order to know the spacing between sets of parallel planes (and to know the angles between the planes in any system except the cubic system, see §22) we must know the physical dimensions of the building blocks of the crystal lattice, i.e., the unit cell dimensions which are "the lattice constants." In the cubic system the units are always cubes, so all that is needed is the length of the edge of the cube a . In other systems, two or more parameters have to be given, in accordance with the list in §3.

The lattice constants for many substances have been determined by x-ray diffraction methods and are given in published tables. Some references where these may be found are given below.

American Institute of Physics Handbook. New York, McGraw-Hill Book Company, 1957.

Donnay, J. D. H., and W. Nowacki. Crystal Data. New York, Geological Society of America, 1954.

Handbook of Physics and Chemistry. Published periodically by the Chemical Rubber Publishing Company, 2310 Superior Avenue, N.E., Cleveland, Ohio.

Pearson, W. B. Handbook of Lattice Spacings and Structures of Metals. New York, Pergamon Press, 1958.

Wyckoff, R. W. G. Crystal Structures. New York, Interscience Publishers, 1960.

7. Values of d from Lattice Constants

The spacing or distance between parallel planes of atoms in a crystal is designated by the letter d :

$$\text{Cubic} \quad \frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

$$d = \frac{a}{(h^2 + k^2 + l^2)^{\frac{1}{2}}} \quad (\text{See §23.})$$

$$\text{Tetragonal} \quad \frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \left(\frac{l}{c}\right)^2$$

$$\text{Hexagonal} \quad \frac{1}{d^2} = \frac{4}{3} \frac{h^2 + hk + k^2}{a^2} + \left(\frac{l}{c}\right)^2$$

$$\text{Orthorhombic} \quad \frac{1}{d^2} = \left(\frac{h}{a}\right)^2 + \left(\frac{k}{b}\right)^2 + \left(\frac{l}{c}\right)^2$$

$$\text{Monoclinic} \quad \frac{1}{d^2} = \frac{h^2}{a^2 \sin^2 \beta} + \frac{k^2}{b^2} + \frac{l^2}{c^2 \sin^2 \beta} - \frac{2hl \cos \beta}{ac \sin^2 \beta}$$

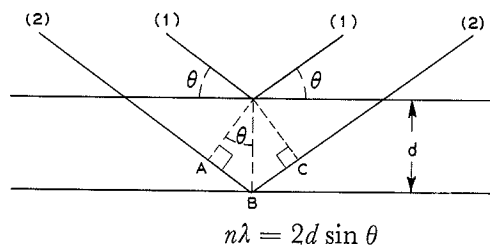
Note that d may be the same for different values of hkl . For example (cubic), (511) and (333). (See §23.) (See Pitfall 4, §24.) For significance of the symbol (333), see §10.

The formulas for the triclinic and rhombohedral systems of axes may be found in C. S. Barrett, *Structure of Metals* (1952).

The following tables give d vs θ or 2θ for various radiations (§9): *Tables for Conversion of X-ray Diffraction Angles to Interplanar Spacing*, (National Bureau of Standards Applied Mathematical Series 10, U.S. Government Printing Office, 1950), for $K\alpha_1$ only, from Mo, Cu, Ni, Co, Fe, Cr; *Tables of Interplanar Spacing, Computed for the Characteristic Radiations of Copper Molybdenum, Iron, Chromium and Cobalt*, H. J. Garrett and R. E. Brocklehurst, Materials Laboratory, Wright Air Development Center, available by number, PB 131764, from the office of Technical Services, U.S. Department of Commerce, Washington 25, D.C., for $K\alpha$, $K\alpha_1$, $K\alpha_2$, $K\beta$.

Alternatively, θ may be determined directly from (hkl) and the lattice constants by formulas given in §9.

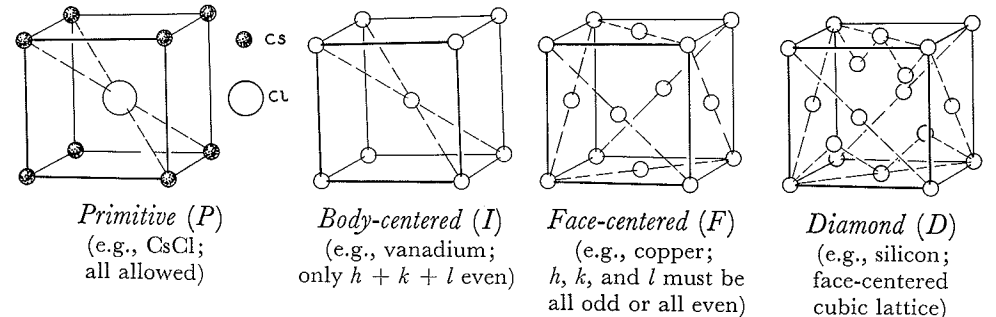
8. X-Ray Reflection from Atomic Planes in a Crystal—Missing Reflections in the Cubic System



When x rays (1) and (2) reflect from atomic planes which are a distance d apart, they will be in phase (and therefore not extinguished) only if the distance traveled by ray (2) is an integral number of wavelengths greater than the distance traveled by ray (1),

so that when it is again traveling with ray (1) it is "in step," i.e., in phase. In the figure, $AB = BC$, $\sin \theta = AB/d$, therefore $AB = d \sin \theta$. If n is an integer and λ is the wavelength of the x rays, $2d \sin \theta$ ($AB + BC$) must equal $n\lambda$ for "reflection" to take place. If a similar plane of atoms is added, just halfway between these two, the extra path length to it will be just half of ABC , the reflected ray will therefore be just out of phase with ray (1) and will cancel it. For this reason body-centered cubic crystals (i.e., crystals with exactly the same atom or grouping of atoms at the center of the cell as at the corners) have no $\{100\}$ reflection; in fact, it turns out that they have only those reflections for which the sum $h + k + l$ (and therefore $h^2 + k^2 + l^2$) is an even number. Note that only $1/8$ of each corner atom lies in the unit cell (see figure, below). There are thus only two atoms in the unit cell of vanadium.

The various cubic lattices for which each reflection is "allowed" are listed under "lattice" in §23. All reflections are allowed for a primitive lattice, which has a unit cell in which no point is exactly like those at the corners of the cell. An example of a structure for each type of cubic lattice is sketched below.



Lattice: The term *lattice* is used by crystallographers to refer to one of the 14 Bravais space lattices (see, for example, C. S. Barrett, *Structure of Metals*, 1952). These are all of the three-dimensional repeat patterns of crystals—every crystal structure is made up of units repeated according to one of these patterns. The lattice points mark the periodicity of the repetition. They do not necessarily mark the positions of atoms.

9. Determination of the Bragg Reflection Angle, θ

Where λ is the wavelength of the x rays used, $\sin \theta = \frac{\lambda}{2d}$ (see §10)

Cubic $\sin^2 \theta = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2)$

Tetragonal $\sin^2 \theta = \frac{\lambda^2}{4} \left[\frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \right]$

Orthorhombic $\sin^2 \theta = \frac{\lambda^2}{4} \left[\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right]$

Hexagonal $\sin^2 \theta = \frac{\lambda^2}{4} \left[\frac{4}{3} \frac{h^2 + k^2 + hk}{a^2} + \frac{l^2}{c^2} \right]$

Alternatively, θ may be found in tables if d is known (see §7).

Wavelengths of Characteristic X Rays from Various Targets (in Ångström units)

	$K\alpha^*$	$K\beta$	β filter	Filter thickness to reduce $K\beta/K\alpha$ to 1/600 (in mm)
Copper	1.542	1.392	Ni	0.021
Chromium	2.291	2.085	V	0.016
Iron	1.937	1.757	Mn	0.016
Cobalt	1.790	1.621	Fe	0.018
Molybdenum	0.711	0.632	Nb or Zr	0.108

* The $K\alpha$ values are the weighted average of two close peaks in the spectrum, $K\alpha_1$ and $K\alpha_2$, the second being one-half the intensity of the first. In nearly all crystal-orienting work, the two will not be resolved.

The β radiation is about one-seventh the intensity of the α radiation and can be greatly reduced, selectively, by use of thin-foil filters containing the elements listed above as β filters. If the β radiation is not filtered out, the reflection of the β radiation may be mistaken for that of the α radiation and misorientation will result.

For determination of missing reflections, see §8 and §11.

10. Orders of Reflection: Notation, Choice

In §9 we are given the equation $\sin \theta = \lambda/2d$. This equation has also been written as $n\lambda = 2d \sin \theta$ (§8). For a given d , various values of θ may satisfy the equation at various integral values of n : 1, 2, 3, 4, etc. The reflections that take place at these various values of θ are known, respectively, as the first-, second-, third-, and fourth-order reflections.

Notation: If we divide through by n , we get $\lambda = 2(d/n) \sin \theta$. Thus, we can think of the second-order reflection from planes with spacing d as being the first-order reflection from planes with spacing $d/2$. The plane which cuts the crystallographic axes at half the length (from the origin) of the intercepts of the (111) plane is the (222) plane (§5). Therefore, the second-order reflection from the (111) plane is commonly called the 222 reflection. Parentheses are omitted to distinguish the reflection from the plane.

Choice: Some orders from a given plane will be stronger than others. These are usually the lower orders, but not always. Some may be missing entirely (§8, §11). Getting more than one order of reflection is sometimes a valuable check (§24). Because of the shape of the sample, a higher angle of reflection may be more convenient, and for this reason it may be necessary to use the higher-order reflections.

For the various orders of reflection from a given plane, θ can be readily found by the equation

$$\sin \theta = n(\lambda/2d).$$

E.g., for NaCl ($a = 5.64$), $d_{(111)} = 3.26$.

For CuK α radiation ($\lambda = 1.54$):

$$\sin \theta_{(111)} = \frac{1.54}{2 \times 3.26} = 0.236$$

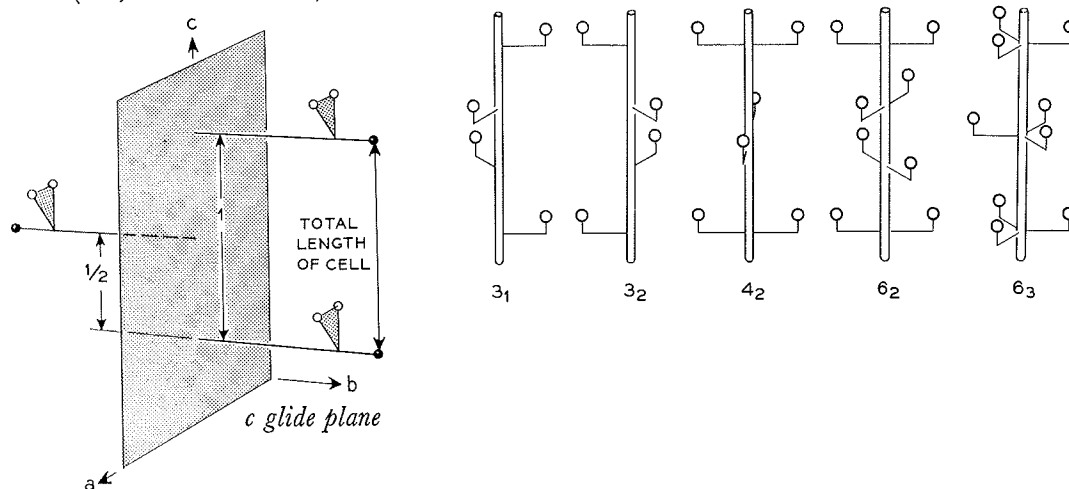
$$\sin \theta_{(222)} = 2 \times \sin \theta_{(111)} = 0.472$$

$$\theta_{(111)} = 13.7^\circ, \quad \theta_{(222)} = 28.2^\circ.$$

11. Space Groups: Glide Planes, Screw Axes, Missing Reflections

When the symmetry of positions of atoms in the crystal structure is considered, two new types of symmetry element must be added to those of §2: glide planes, in which translation is combined with reflection, and screw axes, in which translation is combined with rotation. Examples of these are shown below. Additional screw axes are illustrated in C. W. Bunn, *Chemical Crystallography* (2d ed., 1961).

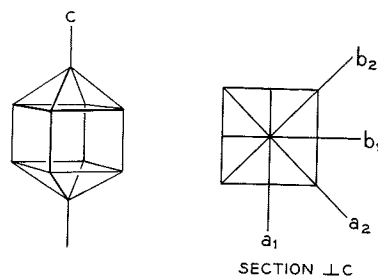
When these additional symmetry elements are included, there are 230 possible ways of combining the symmetry elements. These are called the 230 space groups. Symbols for the space groups give the type of lattice (primitive, body-centered, etc., see §8), the screw axes, and the glide planes and their directions of glide. Thus, the space-group symbol for vanadium, $Im\bar{3}m$, indicates that it is body-centered with point group $m\bar{3}m$ (§4). The space-group symbol for zinc is $P6_3/mmc$. This indicates that the cell is primitive, the principle axis of symmetry is a screw axis 6_3 (see diagram below), and that one of the sets of symmetry planes parallel to this axis is a set of glide planes, with the translation in the c direction (see end of §2). To derive the point-group symmetry from the space-group symmetry, omit the initial letter, omit the subscript if the axis is a screw axis, and change any small letter to m . The above symbol thus becomes $6/mmm$. The physical properties obey the point-group symmetry. However, the point-group symmetry will not tell you about certain x-ray reflections that are missing because of the space-group symmetry. The easiest way to find these is to look up the space group in the *International Tables for X-ray Crystallography* (Volume 1, published by Kynoch Press, Birmingham, England, 1952) where you will find for each space group the conditions that h , k , and l must satisfy in that space group if reflection is to occur. For example, for the space group $P6_3/mmc$, the requirement is that for planes of the type $hh\cdot l$ (like $11\cdot 2$), $l = 2n$ (i.e., l must be even).



12. Visual Examination: Growth Faces, Cleavage Faces, Etch Pits

When a crystal has developed natural *faces* during its *growth*, the symmetry (§4) of arrangement of these faces will usually indicate the location of the crystallographic axes but some cases are ambiguous. The example below illustrates such a case. The correct choice of axes is determined by x-ray diffraction examination.

- Tetragonal example*
- (1) Forms are $\{100\}$ and $\{101\}$;
 - (2) Forms are $\{110\}$ and $\{111\}$;
symmetry $4/m\bar{m}m$.

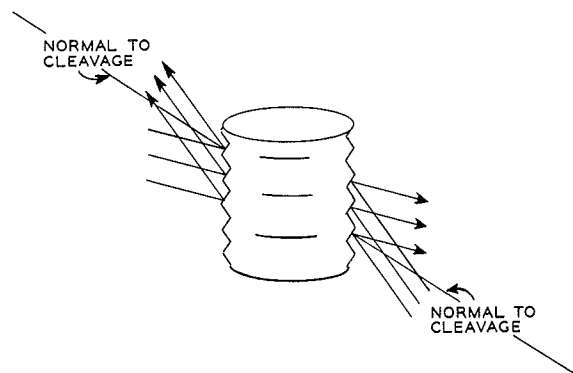


The sizes of faces are determined by growth conditions, and those of one *form* may differ so widely from each other in size as to disguise the symmetry. Angular relationships must of course be maintained (§3), and therefore measurement of interfacial angles will reveal the symmetry in a distorted crystal.

Under certain growth conditions the crystal faces may only develop as small facets on a curved surface. The symmetry of their arrangement can then be determined by light reflection, preferably using parallel rays, as from the sun.

Cleavage faces: may be created by breaking the crystal which can be done if material is plentiful. Unlike some etch pits, and even some growth faces, cleavage faces are always brightly reflecting and very flat, though there may be steps between the flat surfaces. In some cases, as in silicon and germanium, sand-blasting leaves a surface with cleavage facets ($\{111\}$, in this case) which reflect the light and reveal the symmetry. Not every crystal has a cleavage plane. Some have two different planes, some three.

Etch pits: reveal the symmetry because chemical action is the same along all crystallographically equivalent planes, and the faces of a given *form* may thus be the bounding faces of an etch pit. Different etchants (solvents or reactants) may



Reflected light from cleavage facets or etch pit facets gives the orientation of face normals

attack the various faces differently and result in different bounding faces of the pits, but of course the symmetry revealed will be the same. Again, light reflection from the resulting facets is used as above.¹ See C. S. Barrett, *Structure of Metals* (1952), for a fuller discussion of determination of orientation by etch pits, including a table of etchants. Composition faces that are *twin planes* (§13), when known, may also be helpful.

Single crystal check: The cleavage facets or etch facets that are parallel to a particular plane, such as (111) in a crystal, will of course all reflect light at the same angle when the crystal is turned in a bright light. If such reflection from facets is observed from part, but not all, of the sample, held at a given angle to the light, the sample probably includes more than one crystal.

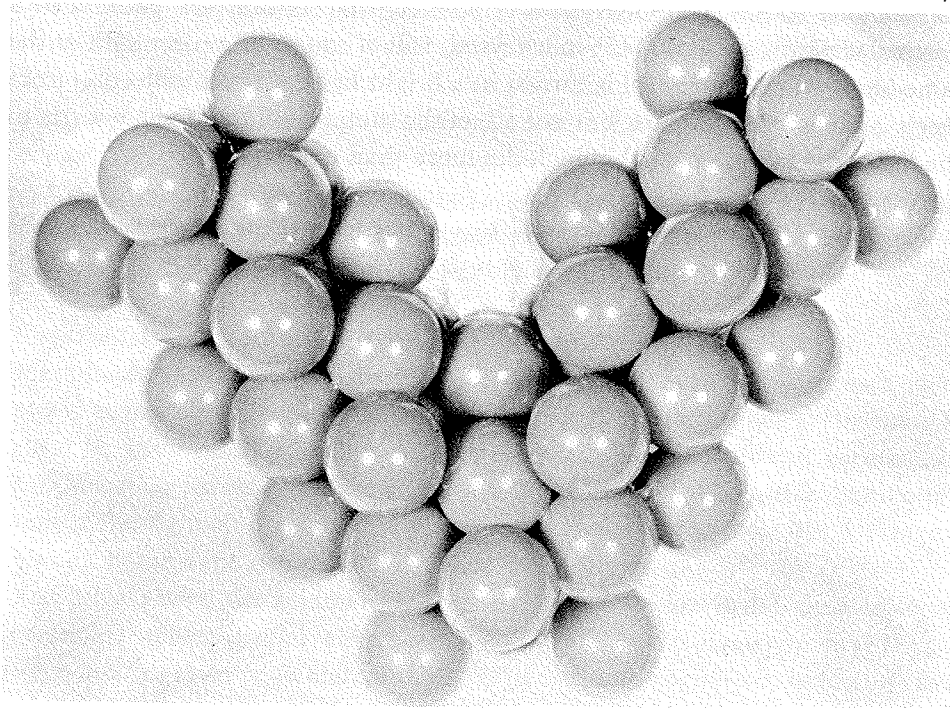
¹ An instrument to aid in the observation of reflected light beams from etched surfaces is made by Micromech Manufacturing Corporation, Union, New Jersey (Optical Orientation System Model WM-OS # 1).

13. Twinning

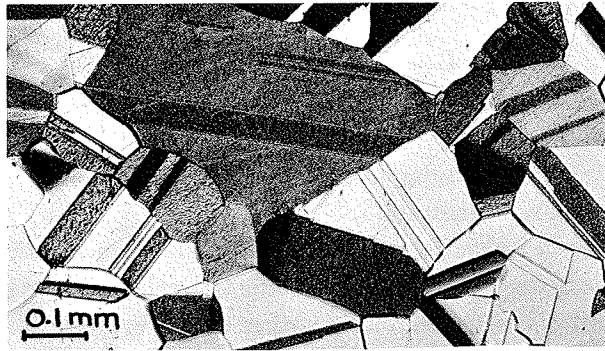
One of the common "mistakes" a crystal may make, in growing, is to change abruptly the orientation of its orderly array of atoms so that the structure of the new part is not related to the old by straight translation, but rather by some crystallographic symmetry element, an axis or plane of symmetry. This phenomenon is known as *twinning*. If the element relating the two parts is an axis of symmetry, it is called the *twin axis*. If it is a mirror plane, it is called the *twin plane*. The surface that separates the two twin-related individuals is called the *composition face*. It may not be planar but it is in most cases and is commonly parallel to the twin plane.

The model shows a face-centered cubic crystal (e.g., copper) "twinned on the (111) plane." The (111) plane is both the twin plane and the composition face. Because this type of twinning is well exemplified by spinel it is called "spinel twinning." A back-reflection Laue photograph of a spinel twin in silicon is shown in Appendix II.

For common examples of twinning "laws" in various crystal systems see E. S. Dana's *A Textbook of Mineralogy* (revised by W. E. Ford, 4th ed., 1932).

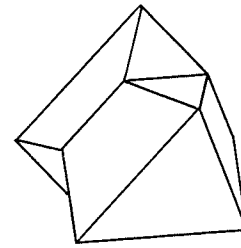


[18]



Polycrystalline alpha brass showing (111) twinning

Polished and etched, $\times 75$. Photomicrograph by F. G. Foster.



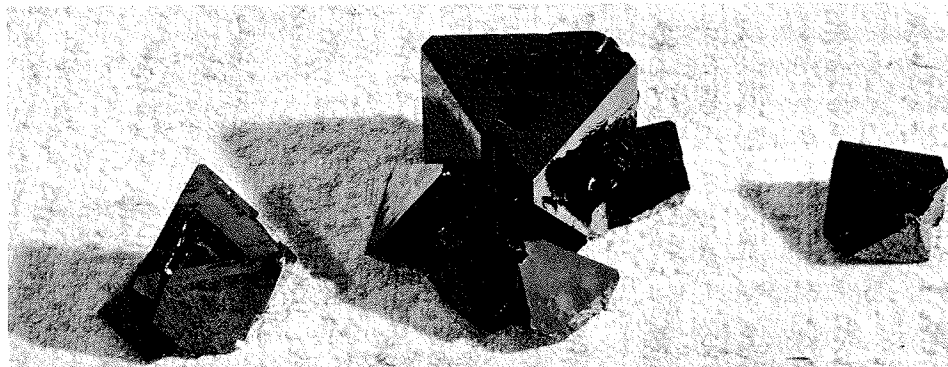
Octahedron (all $\{111\}$ faces), twinned on (111)

Angle between adjacent, twin-related faces is $2 \times 70^\circ 32'$ (see §21).

Etching may frequently be used for the detection of twinning since the etchant acts differently on the differently oriented surfaces. In the photomicrograph the straight boundaries are composition faces between twin-related parts ("twin boundaries"). The curved boundaries are grain boundaries.

In the model of the twinned octahedron, note that the composition face is a mirror plane. Edges of the composition face are parallel to edges in both of the twin-related individuals.

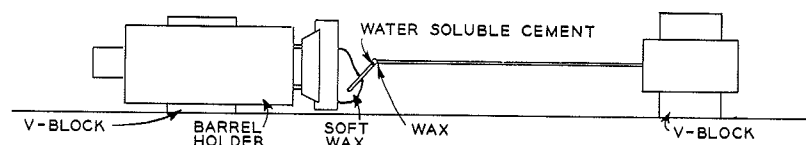
Below are octahedral crystals of $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ grown by J. P. Remeika. Although some have grown together in groups they are not twin-related. The boundary between them is a grain boundary like those in the photomicrograph above.



14. Methods and Materials for Mounting Crystals

It is desirable to orient the crystal approximately by visual examination (§12), if possible, before proceeding to x-ray orientation.

Before attaching the crystal to a holder, consider the ultimate plans for the crystal. Must the adhesive be strong enough to hold the crystal for sawing slices? Will the crystal have to be transferred to some other holder without loss of orientation? One such transfer is illustrated below. (1) A very small crystal



is cemented to the original rod with water-soluble cement. Then the crystal is oriented so that the desired planes are normal to the barrel axes (§15). (2) The long rod for the planned experiment is brought up to the oriented crystal along a straight edge parallel to the barrel axis. The rod is attached to the crystal with hot wax. When this is hard, the water-soluble cement is dissolved and the transfer is complete.

LIST OF USEFUL ADHESIVES

For crystals with high melting points: Quartz wax (mp 150° C), available from Hugh Courtright Company, 7600 Greenwood Avenue, Chicago, Illinois, by the name of #70C Brand thermocouple cement, may be used. Adhesion is adequate for sawing and grinding. The wax is soluble in alcohol.

For crystals with low melting points: Sticky wax (mp 75° C), available by that name from Corning Rubber Company, Inc., 578 Gates Avenue, Brooklyn, New York, may be used. Adhesion is adequate if care is used for sawing and grinding. The wax is soluble in benzene.

For attaching thin plates to a surface, as for dicing: Bayberry wax (mp about 60° C), available from Amend Drug and Chemical Company, 117 East 24 Street, New York, New York, may be used. This wax flows better than sticky wax, but lacks its strength of adhesion. Adhesion is adequate for dicing thin plates if care is used, but not for sawing or grinding. The wax is soluble in benzene.

For easy manipulation: Cheese wax, available from Candy and Company, 35th and South Maplewood Avenue, Chicago, Illinois, by the name of Yellow Cheese wax B-236, may be used. This is a very soft wax, too yielding to hold a crystal for any cutting. The wax is soluble in benzene.

When a crystal has been oriented, held in cheese wax, plaster of Paris may be set around it without disturbing its orientation, to hold it firmly for cutting. In the case of fragile crystals, however, it may be difficult to remove the plaster of Paris later without breaking the crystal. Some prefer Clover Leaf Impression Plaster Double X, quick-setting, available in a wide variety of setting times, water soluble and non-water-soluble. It can be obtained from J. E. Healey Company, 21 Baldwin Street, Newark, New Jersey.

For a water-soluble cement: Any of several substances which form a fine polycrystalline body when they come out of solution may be used. One such substance is Rochelle salt.

For crystals that are soluble in benzene, so that excess wax cannot be removed with benzene: Phenyl salicylate (mp 45° C) may be used (also called "salol"). This is soluble in CCl₄ or trichlorethylene and is available in drug stores. Duco cement, which is soluble in acetone, or Ambroid, soluble in nitrobenzene, may also be used in some cases.

15. The Bond Adjustable Barrel Holder¹

A useful holder which facilitates the x-ray orientation of single crystals has been designed by W. L. Bond of the Bell Telephone Laboratories. The problem it handles is that of x-ray-adjusting the crystal to the desired orientation relative to the holder and maintaining this orientation through the next step of cutting or remounting. This is achieved by mounting the crystal (as described below) on the end of a steel cylinder $1\frac{1}{2}$ in. in diameter and roughly 4 in. long which is held, during x-ray reflection, in a V-block mounted on the x-ray goniometer (§16). This holds the cylinder axis normal to the x-ray beam for a goniometer reading of zero.

After x-ray adjustment, transfer of the barrel holder to a similar V-block mounted on a surface grinder, saw, etc., makes possible a cut of known crystallographic orientation. Alternatively, the crystal may be transferred without loss of orientation to some other holder for polishing or for measurement if both holders are V-block supported and the V-blocks are co-aligned with a straight edge (§14). The crystal is attached (e.g., with wax) to a detachable plate (a) on the end of the cylinder which can be adjusted through small angles relative to the cylinder. When the barrel holder is laid in the V-block which is mounted (as described above) on the goniometer, the goniometer reading gives the angle between the x-ray beam and a plane normal to the cylinder axis. If the desired plane (hkl) is *nearly* normal to the cylinder axis, x-ray reflection will take place when the goniometer reading is *near* θ_{hkl} . When the plane has been adjusted to be exactly normal to the cylinder axis, x-ray reflection will take place when the goniometer reading is exactly θ_{hkl} . This is more fully discussed in §16.

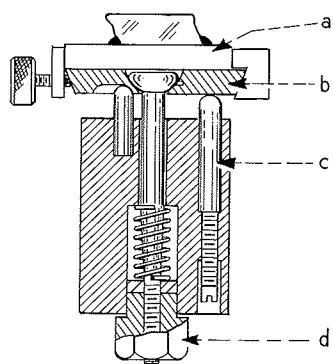


Fig. 1

The adjustment of the spring-held crystal-holding plate relative to the face of the cylinder is achieved by means of three pins, two adjustable (c), one fixed (Fig. 1). Rotation around two mutually perpendicular axes both normal to the barrel axis permits correction of the two angular components of position. Although the crystal could be attached directly to plate b, it has been found more convenient to have removable plates like a which hold

¹ J. G. Walker, H. J. Williams, and R. M. Bozorth, *Rev. Sci. Instr.* 20: 947, 1949.

the crystal. The tension of the spring is controlled by a nut (*d*). When the desired orientation has been achieved, the nut is tightened until its shoulder rests on the end of the barrel, thus rigidly securing the crystal holder for subsequent operations.

A modification of this barrel holder with which the crystal can be held for grinding on a simple lap, has been described by W. L. Bond (Fig. 2).² His diagram of the holder is repeated here.

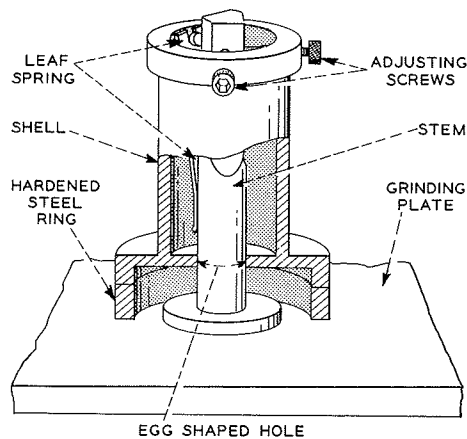


Fig. 2

Transfer to such a holder from a Supper goniometer head³ is illustrated in Fig. 3.

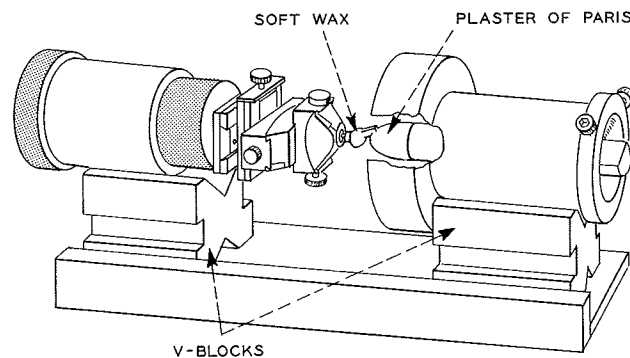


Fig. 3

² W. L. Bond, *J. Sci. Instr.* 38: 63, 1961.

³ Charles Supper Company, Inc., 36 Pleasant Street, Watertown 72, Massachusetts.

16. The X-Ray Goniometer

If a surface whose orientation is known more closely than $\pm 2^\circ$ is needed, and a crystal face or cleavage face cannot be used, then the x-ray goniometer will be needed.¹

The x-ray goniometer is an instrument comprising a set of collimating slits through which the incident x-ray beam travels, a crystal holder which permits rotation of the crystal in the beam around an axis normal to the beam, and an x-ray detector which can be positioned to receive the beam. Figure 1 is a

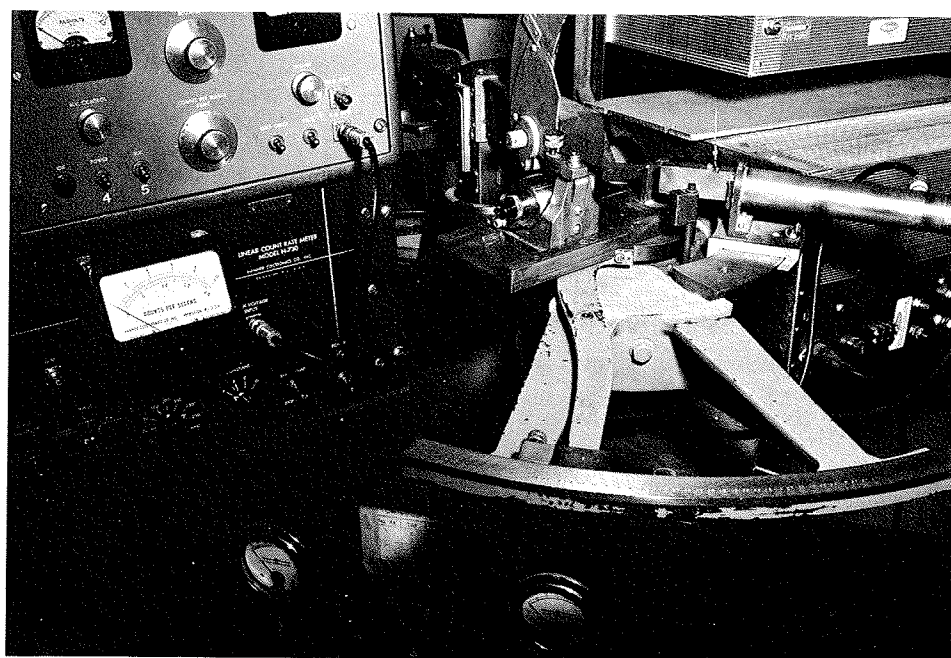


Fig. 1

photograph of such an instrument. From §8 we know that x-ray reflection from a set of atomic planes with interplanar spacing d will occur when x rays of wavelength λ make the Bragg angle θ with the planes such that $n\lambda = 2d \sin \theta$,

¹ If the specimen contains regions which differ from each other in orientation by less than a quarter of a degree, this may not be detectable even by Laue photography or with the regular x-ray goniometer. If such low-angle grain boundaries would be intolerable in the use for which the crystal is being prepared, then a double-crystal x-ray goniometer should be used. See W. L. Bond, *Proc. I.R.E.* 38: 886, 1950, and W. L. Bond and J. Andrus, *Am. Mineralogist* 37: 622, 1952.

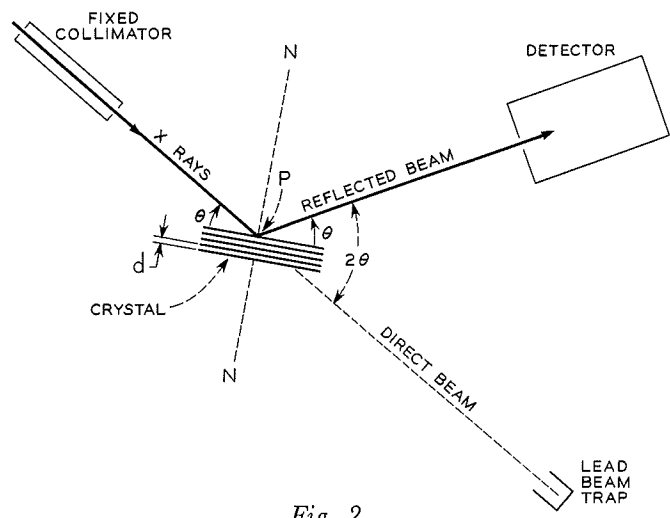


Fig. 2

where n is any integer. The reflected x rays can be detected with an ionization chamber, Geiger counter, or other suitable detector.

In Fig. 2, the x rays are shown passing through the fixed collimator, falling on the (greatly magnified) crystal planes at the angle θ , reflecting from them at the same angle θ into the detector placed at an angle 2θ to the direct beam. Both the detector and the crystal rotate about the instrument axis which is normal to the paper at point P in Fig. 2. The surface of the crystal must be at this axis. The direct beam is normal to this axis and passes through it.

The angle that the axis of the detector makes with the direct beam is read on the "detector scale" (Fig. 3). The angle that the plane normal to the barrel axis makes with the direct beam (in the plane of the paper) is read on the "crystal scale."

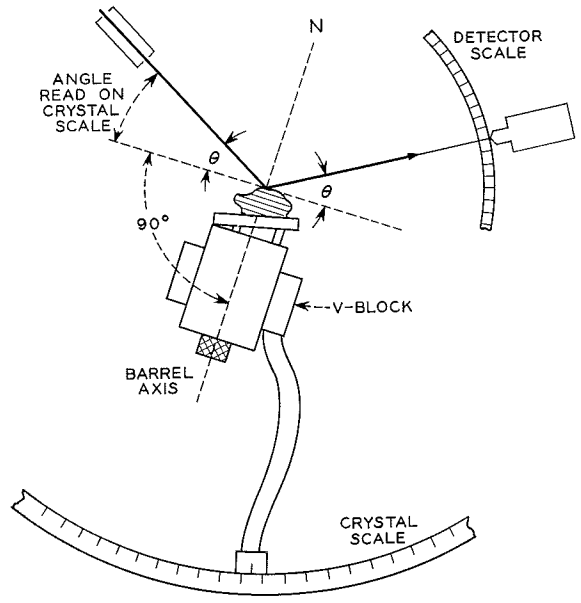


Fig. 3

If the detector is placed at a given angle $2\theta_1$ to the direct beam (Fig. 2), then it will indicate maximum intensity of the x-ray beam when the normal (N) to a set of atomic planes with spacing d_1 bisects the $(180^\circ - 2\theta_1)$ angle between the incident beam and the detector, provided $n\lambda = 2d_1 \sin \theta_1$. E.g., with a copper target tube we wish to get reflection from (111) of silicon. Checking §26, we find that 111 is an allowed reflection for the diamond structure. From §9, or from §7 and d vs θ tables, using $a = 5.43$ for silicon, we find $\theta_{(111)} = 14.2^\circ$. With the x-ray detector set at 28.4° from the direct beam as in Fig. 2, maximum reflected intensity will be indicated when the normal (N) to the (111) planes of the silicon specimen bisects the $(180^\circ - 2\theta)$ angle between the incident beam and the detector. This will occur when these planes are parallel to the axis at P and make the Bragg angle 14.2° with the incident beam.

The Bond barrel holder (§15) makes it possible to achieve this condition and, if desired, generate a surface parallel to these planes. Figure 3 shows diagrammatically the barrel holder in place in a V -block on the goniometer. This V -block is precision-mounted so that the axis of a barrel holder lying in it passes through the instrument axis at P and the goniometer arm is at zero on the crystal scale when the axis of the barrel holder is normal to the x-ray beam. Thus the angle read on the crystal scale is always the angle between the incident beam and a plane normal to the axis of the barrel holder. The aim is to adjust the crystal so that the desired atomic planes are normal to the barrel axis. The barrel holder can then be transferred to a precisely oriented V -block in a grinder or saw and a surface generated normal to its axis and therefore parallel to the desired planes.

The adjustment of the crystal can be systematically achieved as follows. It is assumed that visual examination or Laue photography has already given the orientation within a few degrees so that the specimen can be cemented to the barrel holder with the normal to the desired planes oriented approximately parallel to the barrel axis.

Figure 4 shows the specimen and part of the holder at this stage. The normal to the atomic planes (N) makes a small angle with the barrel axis which may be thought of as having two components, one parallel to the paper in Fig. 4, δ_1 , and the other normal to the paper, δ_2 . In Fig. 4 the angle read on the "crystal scale" of the goniometer when x-ray reflection occurs (as shown by the detector) will not be the Bragg angle θ , but, in this case, $\theta - \delta_1$. If the barrel holder is now rotated 180° around its own axis² and the angle for maximum reflection again read on the "crystal scale," it will be found to be $\theta + \delta_1$.

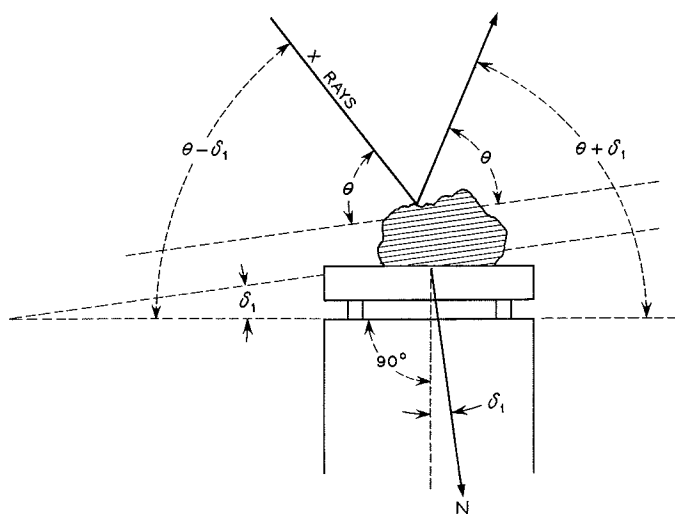
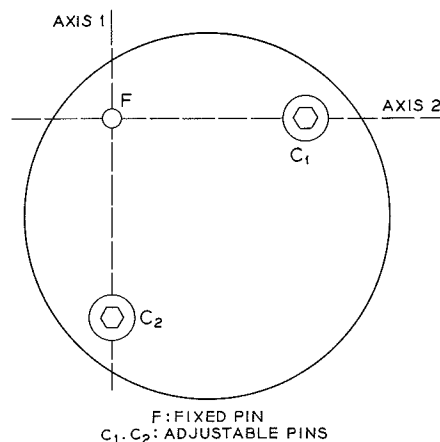


Fig. 4



F: FIXED PIN
C₁, C₂: ADJUSTABLE PINS

Fig. 5

Half of the difference between the two readings gives δ_1 , the angle through which the crystal must be rotated around the instrument axis (normal to the paper in Figs. 2-4). This can be done by adjusting pin C_1 of the Bond barrel holder as described in §15, provided the holder has been so placed in the V-block that axis 1, Fig. 5, is parallel to the instrument axis. (The "instrument axis" is the axis around which the moving parts of the instrument rotate. In Fig. 1 it is normal to the paper at the point P .)

To check the other component, δ_2 , it is only necessary to rotate the barrel holder around its own axis to a position 90° from the initial (0°) position and repeat the process, i.e., read $\theta + \delta_2$ on the crystal scale for maximum reflection in intensity in the "90° position" and then rotate the barrel 180° around its own axis and, finding the maximum reflection intensity, read $\theta - \delta_2$ on the crystal scale.

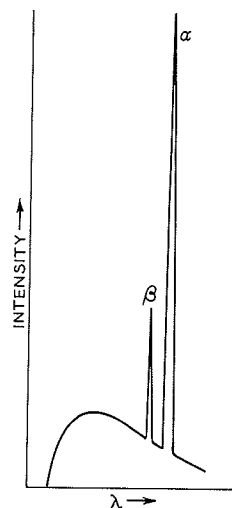
If desired, the goniometer arm indicator may be placed halfway between the " $\theta + \delta$ " and " $\theta - \delta$ " readings on the crystal scale and clamped. The appropriate adjusting pin is then used to rotate the crystal to the position of maximum reflection. If the average of the two readings ($\theta + \delta_1$ and $\theta - \delta_1$) is not the θ calculated for the desired planes, then (a) the instrument has a zero error; or (b) the reflection is not coming from the planes it is thought to be coming from; or (c) the β radiation, rather than the α radiation from the tube, is the reflected ray; or (d) the planes are not parallel to the instrument axis.

² Care must be exercised not to shift the barrel *along* its own axis during this operation since the crystal face must remain at P , Fig. 1. A suitable back stop may be used to prevent this.

17. How to Take Back-Reflection Laue Photographs

Back-reflection Laue photographs are preferred to forward Laue photographs for two reasons: the sample orientation is more easily determined from them and they can be taken with a sample of any thickness and almost any shape.

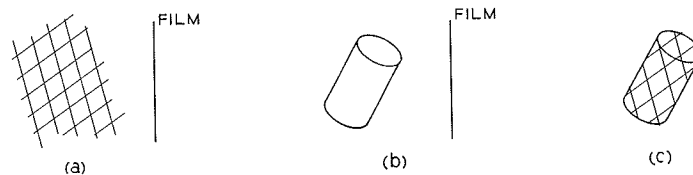
Choice of tube: The x rays from each tube have a range of wavelengths and vary in intensity with wavelength λ , as shown in the figure. The wavelengths at which the β and α peaks occur are characteristic of the particular target metal used and are therefore called *characteristic radiation*. The continuous background, by analogy to white light with its continuous range of wavelengths, is called *white radiation*. Laue photographs require white radiation (§18). At a given voltage and tube current, the tungsten tube gives the most white radiation, and is therefore best for the Laue photographs. Other tubes are poorer but can be used. With a tungsten tube operated at 40 kv, 25 ma, a specimen to film distance of 3 cm, and a collimator opening of about 0.02 in., typical exposure time for a back-reflection Laue photograph would be 25 minutes.



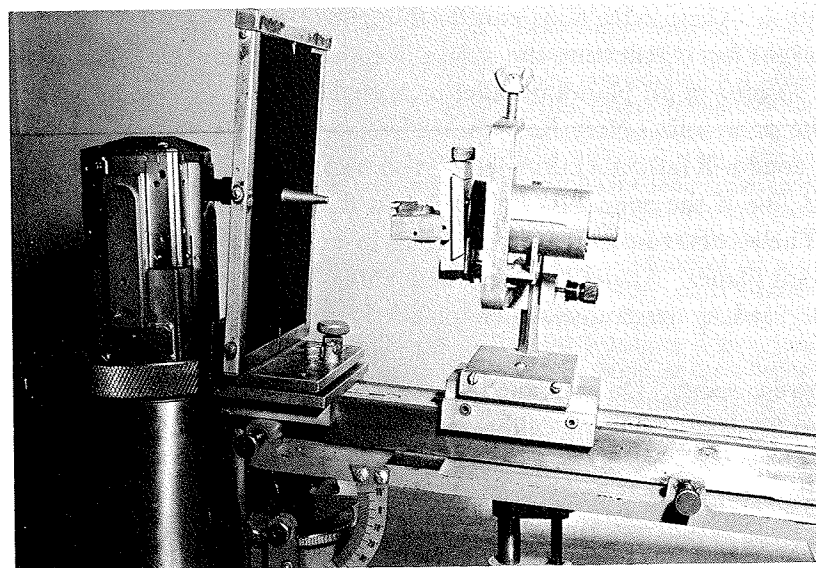
Specimen-to-film distance: This determines the scale of the photograph. A transparent Greninger net for angle measurement (§19) is available from N. P. Nies, 969 Skyline Drive, Laguna Beach, California. This net is for a specimen-to-film distance of 3 cm. The Bond modification of the Greninger net is printed in §19. This net is for a specimen-to-film distance of 5 cm. If use is to be made of either of these, the specimen-to-film distance should be appropriately controlled.

A shorter distance results in shorter exposure time and more spots on the film. A longer distance permits more accurate angular measurement (§19) and facilitates study of the texture of the spots which gives some information about crystal perfection (Appendix II).

Undesirable background: When this results from scattered radiation or fluorescent radiation (if the specimen is just below the tube-target material in the periodic table) it can sometimes be reduced by the insertion of one or two sheets of aluminum foil on top of the film. Kitchen grade is satisfactory but wrinkles must be avoided.



Orientation of the sample relative to the film: This orientation must be accurately known. The Laue photograph gives the orientation of the crystal lattice of the sample relative to the film (a). The orientation of the physical sample relative to the film (b) must be known in order to know the orientation of the crystal lattice relative to the sample (c), which is what is sought. It may be preferable to begin by mounting the crystal on the barrel holder (§15), if it is planned to complete the orientation on the goniometer (§16) after the preliminary Laue photograph. In that case a *V*-block will be needed whose orientation relative to the film is accurately known. The film will give the orientation of the crystal as it was on the x-ray machine (mark the upper right corner of the film so as not to invert or reverse it). It is necessary to ensure in some way that this orientation is not lost when the crystal is removed from the machine.



An x-ray tube, a simple back-reflection Laue camera, and a specimen on a Bond barrel holder suitably mounted for a Laue photograph

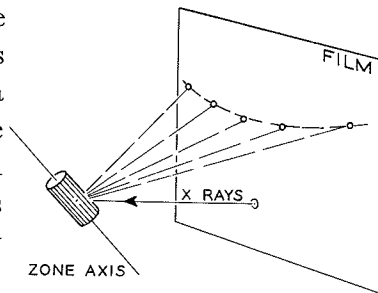
The x rays pass through a hole in the 5 × 7 in. film which is protected by black paper, through the collimator which restricts the beam, and fall on the specimen, from which they are diffracted ("reflected") back onto the film. Back-reflection Laue cameras are available from commercial suppliers of x-ray diffraction equipment.

18. How to Interpret Back-Reflection Laue Photographs

See Appendix II for illustrations.

X-ray reflection occurs when $n\lambda = 2d \sin \theta$ (§7, §8, and §9) where $n =$ an integer, $\lambda =$ wavelength of x rays in Ångström units, $d =$ spacing between atomic planes (also in Å), and $\theta =$ angle of reflection. The d is fixed by the crystal. When the crystal is not moving, θ is fixed. Each plane therefore chooses the right λ from the white radiation for reflection. A plane that happens to reflect radiation of a wavelength that is strong in the incident beam (see beam spectrum in §17) will give a strong spot even though it is not an "important" plane. If it is due to the characteristic radiation, such a spot will be extremely strong. If a group of symmetrically equivalent planes all reflect the characteristic radiation, then the symmetry axis relating them is parallel to the incident beam. In general, each spot on a Laue photograph comes from a different λ . These reflections can be thought of as though the atomic planes were bright facets reflecting the x rays like visible light back to the film.

A *zone* consists of all planes parallel to a single line, the zone axis. Note how the reflections from a zone will fall on a curve which will be a straight line if the zone axis is parallel to the film. These curves intersect in reflections from important planes. Angles between these planes may be used to determine which planes they are.



Angle measurement: On a Laue photograph this is accomplished with the *Greninger net* (§22). This is calibrated to read the angles of normals of planes giving the reflections. Spacing between lines is 2° .

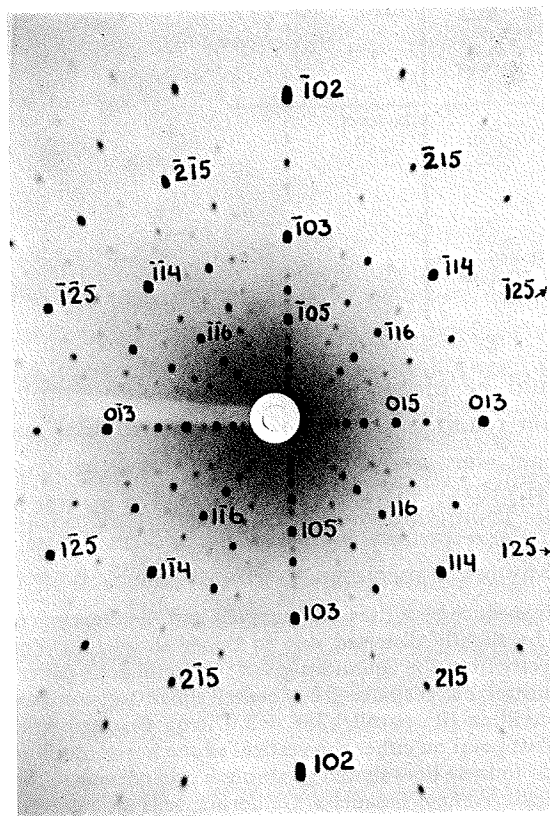
The symmetry of the pattern of reflections from the crystal is of course consistent with the symmetry of the crystal. If the x-ray beam looks down a 4-fold axis of the crystal, the Laue photograph will have a 4-fold axis of symmetry. Since x-ray reflection is the same from either side of a set of atomic planes, the x rays "add a center of symmetry" to the symmetry of the crystal. The symmetry of a crystal with a center of symmetry added is called the *Laue symmetry*. In the point-group chart (§4) the Laue symmetry of any crystal is shown by the first

diagram below its point group in the chart that has a center of symmetry (letter *c* in upper right corner).

To bring an important symmetry axis to the center of the film: When you think you have found such a direction, away from center, it may help to attach a short stick to the specimen with wax, about where you believe the axis to lie. Then turn the specimen until the axis aims along the beam.

Angle measurements between spots on Laue photographs, together with a knowledge of interplanar angles (§21 for cubic crystals, §22 for other systems) form a powerful tool for determining crystal orientation. (See §20.)

For a fuller discussion of the use of Laue photographs for determining crystal orientation, see C. S. Barrett, *Structure of Metals* (1952), page 185. Appendix II shows examples of some back-reflection Laue photographs.



Back-reflection Laue photograph of a single tungsten crystal

Beam approximately along [001]. Specimen-to-film distance 5 cm. Spots labeled with the Miller indices of the planes responsible for the reflected beams producing the spots. The {103} and {114} spots were identified from Greninger-net angle measurements (§19) and comparison with the cubic angle table (§21). The {125} spots were identified (as many others could be) by zonal relationships as follows. Spots (planes) (103) and (114) belong to a zone [*uvw*] which can be found, as described in §5, to be $[\bar{3}\bar{1}1]$. Similarly, spots (114) and (013) define zone $[1\bar{3}\bar{1}]$. The spot (plane) common to both these zones can then be found, as described in §5, to be (215). (Note that all signs may be reversed and still refer to the same planes of atoms.) Size of original photograph: $4\frac{1}{4} \times 6\frac{1}{2}$ in.

19. The Greninger Net¹

The location of each spot on a back-reflection Laue photograph indicates the orientation of a set of atomic planes relative to that of the direct beam. If M is the distance along the film from the point where the direct beam passed through it (usually, though not always, precisely at the center of the hole) to the point where the reflected beam made the spot, and the specimen-to-film distance is R , then $M/R = \tan (180^\circ - 2\theta)$ and the angle between the atomic plane normal and the direct beam is $90^\circ - \theta$ (see Fig. 1). Clearly, from this a net could be

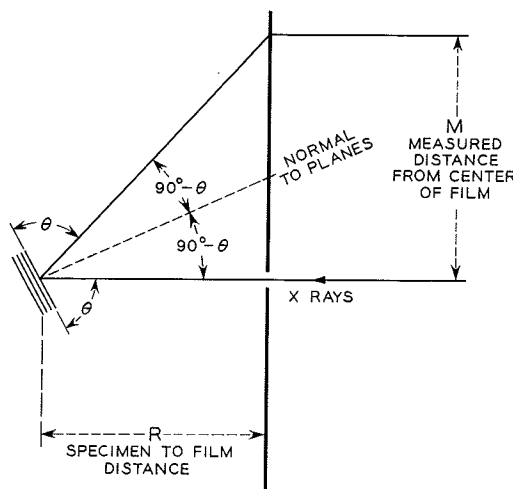


Fig. 1

made for any given R which could be placed over the Laue photograph, making possible angular orientation readings without repetitious calculation. The data for constructing such a net were given by A. B. Greninger in *Zeitschrift für Kristallographie*, Volume 91 (1935), page 424, and a copy of his net is given, scaled 1:1, in §19. This net is for a specimen-to-film distance of 3 cm. It will of course give wrong readings for any other specimen-to-film distance.

¹ Although an effort has been made to reproduce both the Greninger net and the Greninger-Bond net without distortion, they may be slightly distorted due to paper shrinkage. For greater accuracy, nets may be drawn directly from the equations given in Greninger's paper. Check points for the two nets are: *Greninger* net distance from center down to meridian $32^\circ = 6.15$ cm, distance from center to meridian 10° , parallel $28^\circ = 5.07$ cm; *Greninger-Bond* net distance from the center to the outermost point on either the vertical or the horizontal line through the center = 6.4 cm. A line at 45° to these lines should go through grid corners to the outermost corner. N. P. Nies, 1495 Coolidge Avenue, Pasadena, California, sells a Greninger net for a 3 cm specimen-to-film distance that is not measurably distorted.

There are several ways in which this net is useful. The Laue photograph must be placed with the "direct-beam point" (as defined above) at the center of the net for all measurements.

In the following paragraphs we will speak of the "angle between two spots" as a short-cut expression for the "angle between the normals to the planes responsible for the diffracted beams which made the two spots." The Greninger net is so calibrated that angles read between spots or between a spot and the direct beam give directly the angles between plane normals or between a plane normal and the direct beam (see Fig. 2).

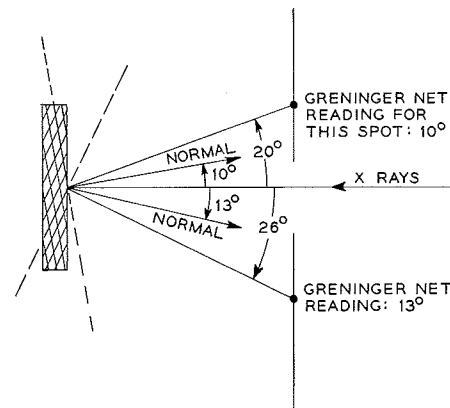


Fig. 2

The angle between the direct beam (center) and any spot: This may be read along either of the two mutually perpendicular straight lines by placing the spot on the line and reading the net angle between the spot (calibrated as the atomic-plane normal) and the center. If a line is drawn on the film through the center, indicating the direction in the film that was vertical when the photograph was taken, then the position of this on the calibrated circular arc supplied with the Greninger net will indicate the "azimuth" in which the atomic plane normal lies.

In the lower half of the diagram on page 35 the lines running up and down the page are parallels; those running across the page are meridians. There is an important difference in the way these two sets of lines are used (see the following paragraph, and paragraphs 3 and 4 of "Uses of the Stereographic Projection," §25).

Angles between spots in a zone: These can be read from the net. A row of spots belonging to one zone (§18) will lie parallel to one of the meridian lines of the

Greninger net and the angles between the spots are read along (or in a direction parallel to) a meridian. This gives us a very powerful tool for identifying spots on the film, as illustrated in §20.

Caution: Readings along the "parallels" do not give correct angles between spots.

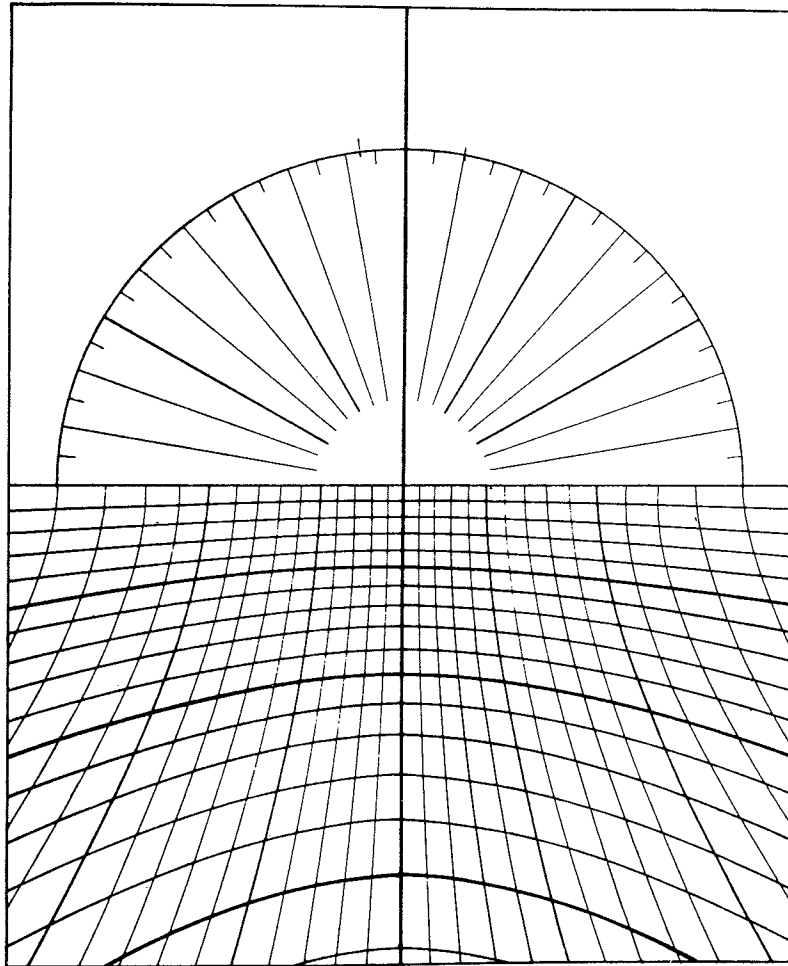
The latitude and longitude of a spot: Relative to the direct beam as 0° , 0° , the latitude and longitude of any spot can be read from the net. This use is of special value in conjunction with the stereographic projection and is therefore explained in the section on "Uses of the Stereographic Projection" (§25).

THE GRENINGER-BOND NET

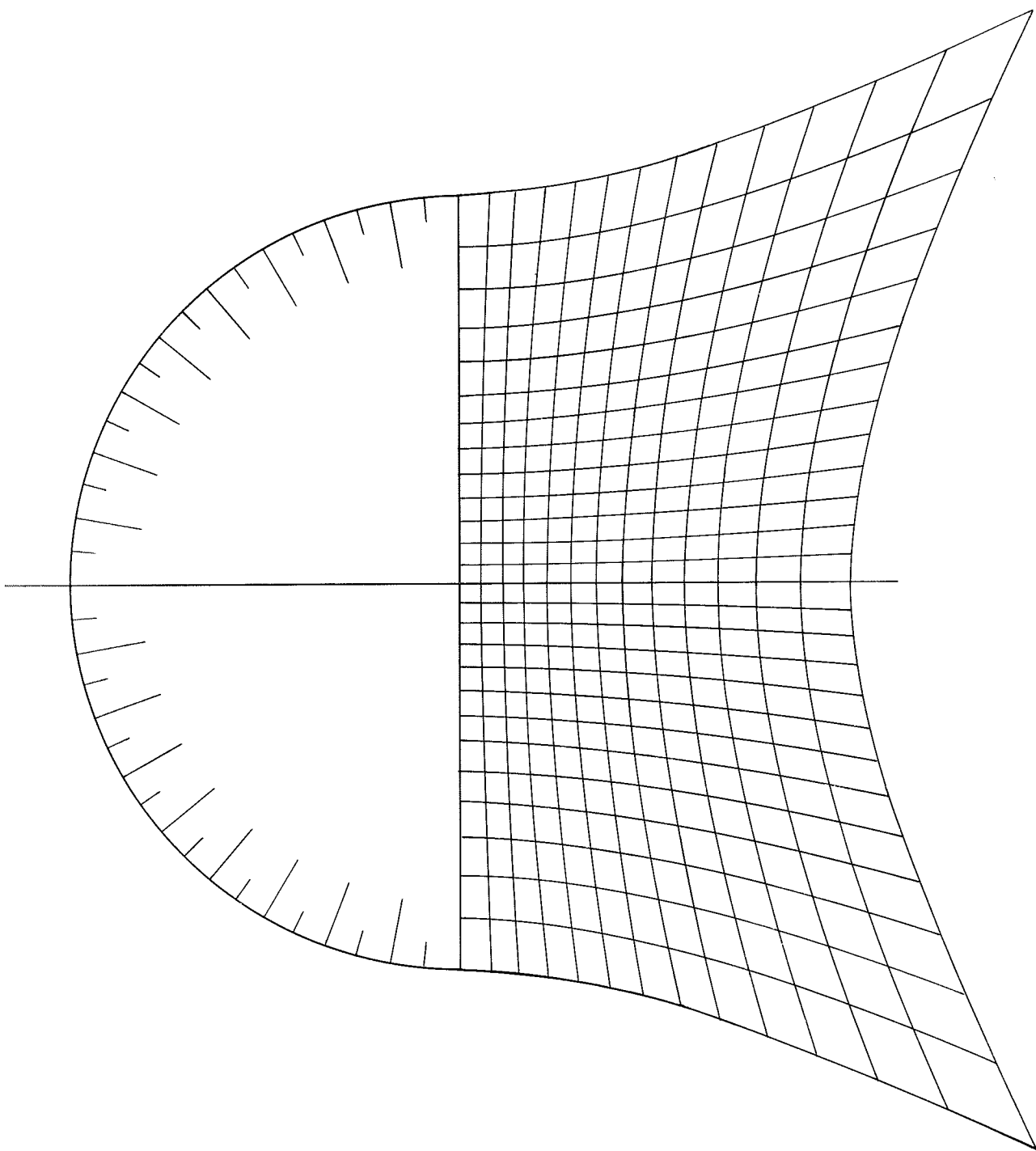
In the Greninger net as modified by Walter L. Bond, both sets of lines are meridians, that is, angles between spots may be read along either the lines that are approximately vertical (running up and down the page) on this chart or the lines normal to these. As given here, this chart is correct for a 5 cm specimen-to-film distance.

While the Greninger-Bond net cannot be used for determining the latitude and longitude of a spot, it has the advantage that all its curves are zonal curves, i.e., curves along which spots belonging to a given zone will lie. The angles at the intersection of any pair of these are those that would be made between rows of spots from two zones normal to each other. This feature of the Greninger-Bond net has been used in §20.

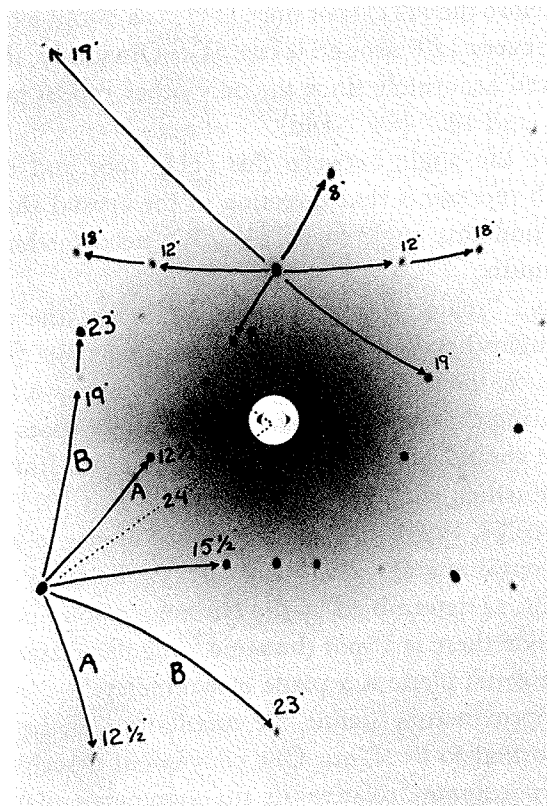
As in the Greninger net, the divisions are at 2° intervals.



THE GRENINGER NET



20. Example of the Use of a Back-Reflection Laue Photograph and Stereographic Projection to Determine the Approximate Orientation of the Direction in a Germanium Crystal That Is Normal to the Film



Examination of the Laue photograph showed a "major spot" at the lower left which was not only a strong reflection but one through which many zones¹ passed. To test whether this was a reflection from a plane whose normal was a 2-fold axis [the (110) plane] or a 3-fold axis [the (111) plane] or a 4-fold axis [the (100) plane], the Greninger net was used for angular measurements along several zones to neighboring spots. The results, shown in the figure, indicated

¹ More properly, "rows of reflections from crystallographic planes. All planes reflecting spots that lie in one row belong to one zone."

that zones B, B were symmetrically related to each other (were "crystallographically equivalent") as were also zones A, A . The angle between a symmetrically related pair could be roughly estimated by placing a zone along a suitable curve on the Greninger-Bond net (remembering always to keep the center of the net at the center of the photograph) and seeing where the symmetrically equivalent zone lay. The angle was found to be greater than 90° and less than 135° ($90^\circ + 45^\circ$).

This spot is therefore the (111) spot since $[111]$ is a 3-fold axis with symmetrically related zones every 120° around it (see §4). Obviously, the angle does not need to be measured accurately since the only other repeat possibilities would be 90° (for 4-fold) and 180° (for 2-fold).

We can measure the angle between this (111) spot and the center of the photograph (which represents that direction in the crystal that was normal to the film) and we find this angle to be 24° . We need further information to determine the azimuth.

There is a second "major spot" above the center of the photograph, and measurement of angles between this and neighboring spots (see figure) shows this to be a 2-fold axis, the reflection from the (011) plane. A further check can be made by measuring the angle between the two major spots which we find to be about 35° , as it should be for the angle between $[111]$ and $[011]$, written " $[111] \wedge [011]$." Further, the plane in which $[111]$ and $[011]$ lie should be a symmetry plane (see §4, §22) and measurement of angles to a few spots either side of this zone containing the (111) and (011) reflections (measured along zones normal to this, as determined by the Greninger-Bond net) shows that for every spot on one side there is a spot the same "angular distance" away on the other side, i.e., the zonal plane is a plane of symmetry.

We can now approximately define the orientation of the direction in the specimen that is normal to the film. One convenient description would be to give its angular coordinates relative to the symmetry plane and axis just determined. It lies about 8° away from a direction in the plane that makes an angle of 24° with the $[111]$ direction, measured toward $[011]$.

There is no need to say which side of the symmetry plane it lies on, since directions related by the symmetry plane are crystallographically equivalent.

By superposing the stereographic projection of the major planes of a cubic crystal (§25) on the stereographic net (§25), we can determine approximately where the direction in question lies and find that it is close to $[123]$, i.e., close to the normal to the (123) plane.

The spot immediately below the center hole in the film is in the right angular relationship to (011) and (111) spots to be the (123) spot.

From an approximate orientation such as this we can rotate the crystal until it is approximately in the desired orientation, check it again with a Laue photograph, and then proceed to orient it more accurately with the aid of the x-ray goniometer (§16).

21. Angles between Planes in Crystals of the Cubic System

(R. M. Bozorth, *Phys. Rev.* 26:390, 1925)

$\{h_1k_1l_1\}$	^	$\{h_2k_2l_2\}$	All values of the angles between $\{h_1k_1l_1\}$ and $\{h_2k_2l_2\}$						
100	^	100	0°	90°					
		110	45°	90°					
		111	54°44'						
		210	26°34'	63°26'	90°				
		211	35°16'	65°54'					
		221	48°11'	70°32'					
		310	18°26'	71°34'	90°				
		311	25°14'	72°27'					
		320	33°41'	56°19'	90°				
		321	36°42'	57°42'	74°30'				
		110	^	110	0°	60°	90°		
111	35°16'			90°					
210	18°26'			50°46'	71°34'				
211	30°			54°44'	73°13'	90°			
221	19°28'			45°	76°22'	90°			
310	26°34'			47°52'	63°26'	77°5'			
311	31°29'			64°46'	90°				
320	11°19'			53°58'	66°54'	78°41'			
321	19°6'			40°54'	55°28'	67°48'	79°6'		
111	^			111	0°	70°32'			
		210	39°14'	75°2'					
		211	19°28'	61°52'	90°				
		221	15°48'	54°44'	78°54'				
		310	43°5'	68°35'					
		311	29°30'	58°31'	79°58'				
		320	36°48'	80°47'					
		321	22°12'	51°53'	72°1'	90°			
210	^	210	0°	36°52'	53°8'	66°25'	78°28'	90°	
		211	24°6'	43°5'	56°47'	79°29'	90°		
		221	26°34'	41°49'	53°24'	63°26'	72°39'	90°	
		310	8°8'	31°57'	45°	64°54'	73°34'	81°52'	
		311	19°17'	47°36'	66°8'	82°15'			
		320	7°7'	29°45'	41°55'	60°15'	68°9'	75°38'	82°53'
		321	17°1'	33°13'	53°18'	61°26'	70°13'	83°8'	90°
		211	^	211	0°	33°33'	48°11'	60°	70°32'
221	17°43'			35°16'	47°7'	65°54'	74°12'	82°12'	
310	25°21'			49°48'	58°54'	75°2'	82°35'		
311	10°1'			42°24'	60°30'	75°45'	90°		
320	25°4'			37°34'	55°31'	63°5'	83°30'		
321	10°54'			29°12'	40°12'	49°6'	56°56'		
	70°54'			77°24'	83°44'	90°			
221	^	221	0°	27°16'	38°57'	63°37'	83°37'	90°	
		310	32°31'	42°27'	58°12'	65°4'	83°57'		
		311	25°14'	45°17'	59°50'	72°27'	84°14'		
		320	22°24'	42°18'	49°40'	68°18'	79°21'	84°42'	
		321	11°29'	27°1'	36°42'	57°41'	63°33'	74°30'	
	79°44'	84°53'							
310	^	310	0°	25°51'	36°52'	53°8'	72°33'	84°16'	
		311	17°33'	40°17'	55°6'	67°35'	79°1'	90°	
		320	15°15'	37°52'	52°8'	74°45'	84°58'		
		321	21°37'	32°19'	40°29'	47°28'	53°44'	59°32'	
			65°	75°19'	85°9'	90°			

ANGLES BETWEEN PLANES IN CRYSTALS OF THE CUBIC SYSTEM
ALONG SELECTED ZONES

(Low-index spots will, in general, be the stronger ones)

Zone axis [100] from (001) to (011)		Zone axis $[\bar{1}01]$ from ($\bar{1}\bar{1}\bar{1}$) to (111)	
(001) \wedge	(018)	7° 07.5'	($\bar{1}\bar{1}\bar{1}$) \wedge
	(017)	8 08	($\bar{5}\bar{4}\bar{5}$)
	(016)	9 27.5	($\bar{4}\bar{3}\bar{4}$)
	(015)	11 18.5	($\bar{3}\bar{2}\bar{3}$)
	(029)	12 31.5	($\bar{5}\bar{3}\bar{5}$)
	(014)	14 02	($\bar{7}\bar{4}\bar{7}$)
	(072)	15 56.5	($\bar{2}\bar{1}\bar{2}$)
	(013)	18 26	($\bar{5}\bar{2}\bar{5}$)
	(025)	21 48	($\bar{3}\bar{1}\bar{3}$)
	(037)	23 12	($\bar{7}\bar{2}\bar{7}$)
	(049)	23 57.5	($\bar{4}\bar{1}\bar{4}$)
	(012)	26 34	($\bar{5}\bar{1}\bar{5}$)
	(059)	29 03.5	($\bar{6}\bar{1}\bar{6}$)
	(047)	29 44.5	($\bar{7}\bar{1}\bar{7}$)
	(035)	30 58	($\bar{8}\bar{1}\bar{8}$)
	(023)	33 42	(101) \wedge
	(057)	35 32.5	(818)
	(034)	36 52	(717)
	(045)	38 39.5	(616)
	(056)	39 48.5	(515)
	(078)	41 11	(414)
	(011)	45 00	(727)
			(313)
			(525)
Toward [010]			(212)
Zone axis $[\bar{1}\bar{1}0]$ from (001) to (111)			(747)
(001) \wedge	(119)	8 56	(535)
	(118)	10 01.5	(323)
	(117)	11 25.5	(434)
	(116)	13 16	(545)
	(115)	15 47.5	(111)
	(229)	17 26.5	Zone axis $[\bar{1}\bar{1}\bar{1}]$ from (101) to (011)
	(114)	19 28.5	(101) \wedge
	(227)	22 00	(314)
	(113)	25 14.5	(213)
	(338)	27 56.5	(325)
	(225)	29 30	(112)
	(337)	31 13	(235)
	(112)	35 16	(123)
	(447)	38 56.5	(011)
	(335)	40 19	
	(223)	43 19	
	(334)	46 41	
	(556)	49 41	
	(111)	54 44	
Toward [110]			

Note that the sums of indices of two planes ($h_1 + h_2, k_1 + k_2, l_1 + l_2$) give the indices of a plane lying between them in the same zone, e.g., (213) lies between (101) and (112), but (123) lies between (011) and (112).

ANGLES BETWEEN SOME HIGHER-INDEX PLANES
IN THE CUBIC SYSTEM: $h_1k_1l_1 \wedge h_2k_2l_2$

$h_1k_1l_1$	$h_2k_2l_2$			$h_1k_1l_1$	$h_2k_2l_2$		
	(001)	(011)	(111)		(001)	(011)	(111)
014	14° 02'	30° 58'	45° 33.5'	225	29° 30'	30° 30'	25° 14'
015	11 18.5	33 41.5	47 12.5	227	22 00	32 33	32 44
016	9 27.5	35 32.5	48 22	133	46 30.5	13 16	22 00
017	8 08	36 52	49 13	233	50 14.5	25 14.5	10 01.5
018	7 07.5	37 52.5	49 52.5	334	46 41	31 54.5	8 03
025	21 48	23 12	41 22	335	40 19	30 23	14 25
027	15 56.5	29 03.5	44 27.5	337	31 13	30 15	23 31
029	12 31.5	32 28.5	46 27.5	144	45 52	10 01.5	25 14.5
034	36 52	8 08	36 04	344	51 20.5	27 56.5	7 19.5
035	30 58	14 02	37 37	155	45 33.5	8 03	27 13
037	23 12	21 48	40 42	255	47 07.5	15 47.5	19 28.5
045	38 39.5	6 20.5	35 45.5	355	49 23	22 59.5	12 16.5
047	29 44.5	15 15.5	38 01.5	455	52 01	29 30	5 46
056	39 48.5	5 11.5	35 35.5	124	29 12.5	22 12.5	28 07.5
057	35 32.5	9 27.5	36 21	125	24 05.5	25 21	32 35.5
114	19 28.5	33 33.5	35 15.5	134	38 19.5	13 54	25 04
115	15 47.5	35 16	38 56.5	135	32 18.5	17 01.5	28 33.5
116	13 16	36 35	41 28	234	42 02	23 12	15 13.5
117	11 25.5	37 37	43 18.5	235	35 47.5	23 24.5	20 31
118	10 01.5	38 26	44 42.5	245	41 48.5	18 26	18 47
223	43 19	30 58	11 25	345	45 00	25 50.5	11 33.5

This table will be of most help if it is used together with the stereographic projections (§25). For example, note that the information $(014) \wedge (001) = 14^\circ 02'$, implies, by symmetry, the following additional information: $(104) \wedge (001) = 14^\circ 02'$, $(\bar{1}04) \wedge (001) = 14^\circ 02'$ [plus the (hkl) equivalents with $(00\bar{1})$] and further that $(014) \wedge (010) = 90^\circ - 14^\circ 02'$, $(104) \wedge (100) = 90^\circ - 14^\circ 02'$, etc., and further that $(041) \wedge (010) = 14^\circ 02'$, $(401) \wedge (100) = 14^\circ 02'$, etc., and further that $(041) \wedge (001) = 90^\circ - 14^\circ 02'$, $(401) \wedge (001) = 90^\circ - 14^\circ 02'$, etc.

Reference: *Angles between Planes in Cubic Crystals*, R. J. Peavler and J. N. Lenusky, published by Amer. Inst. Met. Eng., 1962, as Inst. Met. Div. Series No. 8.

22. Angles between Planes in the Orthogonal Systems

These and other useful equations, including the equation for the triclinic system, may be found in C. S. Barrett, *Structure of Metals* (1952).

$$\text{Cubic } \cos \phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{[(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)]^{1/2}}$$

$$\text{Tetragonal } \cos \phi = \frac{\frac{h_1 h_2 + k_1 k_2}{a^2} + \frac{l_1 l_2}{c^2}}{\left[\left(\frac{h_1^2 + k_1^2}{a^2} + \frac{l_1^2}{c^2} \right) \left(\frac{h_2^2 + k_2^2}{a^2} + \frac{l_2^2}{c^2} \right) \right]^{1/2}}$$

Hexagonal

$$\cos \phi = \frac{h_1 h_2 + k_1 k_2 + \frac{1}{2}(h_1 k_2 + h_2 k_1) + \frac{3}{4} \frac{a^2}{c^2} l_1 l_2}{\left[\left(h_1^2 + k_1^2 + h_1 k_1 + \frac{3}{4} \frac{a^2}{c^2} l_1^2 \right) \left(h_2^2 + k_2^2 + h_2 k_2 + \frac{3}{4} \frac{a^2}{c^2} l_2^2 \right) \right]^{1/2}}$$

$$\text{Orthorhombic } \cos \phi = \frac{\frac{h_1 h_2}{a^2} + \frac{k_1 k_2}{b^2} + \frac{l_1 l_2}{c^2}}{\left[\left(\frac{h_1^2}{a^2} + \frac{k_1^2}{b^2} + \frac{l_1^2}{c^2} \right) \left(\frac{h_2^2}{a^2} + \frac{k_2^2}{b^2} + \frac{l_2^2}{c^2} \right) \right]^{1/2}}$$

Tables of interplanar angles for certain noncubic substances have been published. Examples of these are: Alan Lawley, *Trans. M. S. of AIME* 218: 956, 1960 (for Mg, Re, Ru, Zn, and Cd), and R. E. Frounfelker and W. M. Hirthe, *Trans. M. S. of AIME* 224: 196, 1962. This paper gives selected $(hkl) \wedge (001)$ angles for tetragonal crystals with $c/a = 0.500$ to 2.00 in increments of 0.050 , as well as the same angles for $\beta - \text{Sn}$, SnO_2 , Pb_3O_4 , and TeO_2 .

23. Allowed Reflections in the Cubic System

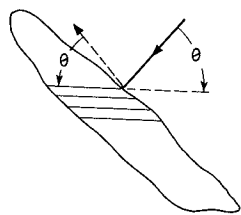
Reflections through $h^2 + k^2 + l^2 = 998$ are given in the *Internationale Tabellen zur Bestimmung von Kristallstrukturen*, Vol. 2 (Berlin, Gebrüder Borntraeger, 1935).

$h^2 + k^2 + l^2$	$(h^2 + k^2 + l^2)^{1/2}$	Allowed, for lattice ¹	{hkl}
1	1.000	P	100
2	1.414	PI	110
3	1.732	PFD	111
4	2.000	PIF	200
5	2.236	P	210
6	2.449	PI	211
7	not allowed		
8	2.828	PIFD	220
9	3.000	P	300, 221
10	3.162	PI	310
11	3.317	PFD	311
12	3.464	PIF	222
13	3.606	P	320
14	3.742	PI	321
15	not allowed		
16	4.000	PIFD	400
17	4.123	P	410, 322
18	4.243	PI	411, 330
19	4.359	PFD	331
20	4.472	PIF	420
21	4.583	P	421
22	4.690	PI	332
23	not allowed		
24	4.899	PIFD	422
25	5.000	P	500, 430
26	5.099	PI	510, 431
27	5.196	PFD	511, 333
28	not allowed		
29	5.385	P	520, 432
30	5.477	PI	521
31	not allowed		
32	5.657	PIFD	440
33	5.745	P	522, 441
34	5.831	PI	530, 433
35	5.916	PFD	531
36	6.000	PIF	600, 442
37	6.083	P	610
38	6.164	PI	611, 532
39	not allowed		
40	6.325	PIFD	620
41	6.403	P	621, 540, 443
42	6.481	PI	541
43	6.557	PFD	533
44	6.633	PIF	622
45	6.708	P	630, 542
46	6.782	PI	631
47	not allowed		
48	6.928	PIFD	444

¹ For explanation, see §8.

24. Examples of Pitfalls—Difficulties Actually Encountered in Crystal Orientation

1. To make a $\{111\}$ plate from (cubic) gallium arsenide, θ for $\{222\}$ was found from a nomograph, using the lattice constant $a = 5.64 \text{ \AA}$. The θ read was 27° . A plane reflecting at 26.9° was found and, since chart reading of angles is only approximate, the plate was cut parallel to this. A Laue photograph showed only a plane of symmetry, no 3-fold axis. Calculation of θ for $\{222\}$ gave 28.3° . The plane with the next lower θ (i.e., the next lower $h^2 + k^2 + l^2$) is $\{311\}$ (§23). Calculation of the θ for this plane gave 26.9° , the angle actually found. A check with a different order of reflection might have caught the error (§9), but an accurate calculation would have avoided it at the start.
2. The unit cell of bismuth is like a cube slightly stretched along the cube diagonal. Only the "stretched diagonal" direction is a 3-fold symmetry axis. But three other directions look very like 3-fold axes in Laue photographs and have planes normal to them whose θ angles are close to, *but not the same as*, that of the plane normal to the 3-fold axis. One of these "pseudo-3-fold" axes was chosen by mistake, again avoidable by accurate calculation of angle.
3. A Laue photograph of a hexagonal crystal showed only a plane of symmetry. It was assumed that the 6-fold axis would lie in this plane of symmetry. (In the class $6/mmm$, six of the seven symmetry planes *do* contain the 6-fold axis.) Reference to tables (§6) however, showed the symmetry of this crystal to be $6/m$. The 6-fold axis was normal to the symmetry plane. Note the importance of knowing the point group of a crystal.
4. A large cylindrical crystal of silicon had its length along the $[111]$ direction. In order to get a plate larger than the cross section of the cylinder, another $\{111\}$ plane was sought, about 70° from the plane of the cross section of the cylinder (§21). The third-order reflection was used in order to get a big enough reflection angle, θ , to avoid having the reflected beam stopped by the specimen itself. A reflection was found at precisely the correct θ angle and several plates were cut. They were later found to have been cut parallel to $\{511\}$, which has precisely the same θ angle as $\{333\}$ (since $h^2 + k^2 + l^2 = 27$ for both, §23). If the other orders of $\{111\}$ had been sought after the first cut, as a check on $\{333\}$, the error would have been discovered. A Laue photograph would also have revealed the error.



25. The Stereographic Projection

INTRODUCTION

The stereographic projection provides a useful way of showing the three-dimensional relationships among planes and directions in a crystal on our two-dimensional piece of paper. An understanding of it is essential for efficient use of the ξ_4 chart. The power of its use as a tool in working with crystals will be demonstrated in the following pages.

The first figure on the opposite page shows a cubic crystal centered in a sphere. From the center, a normal to each face is erected and extended until it meets the sphere in a point. These points and the normals themselves are called "poles" of the faces. This assemblage of points is a *spherical projection* of the faces of the crystal. It would look the same even if growth conditions had caused the crystal to be elongated or flattened in one direction. It thus extracts from the crystal the important information concerning its faces, namely, their angular relations to each other.

To transfer this information in useful form to our two-dimensional paper we need one more step. In the second diagram a line connects the $(00\bar{1})$ point of the spherical projection to each point on the opposite half of the sphere. Where these lines cut the plane on which the opposite half of the sphere is based, they produce a set of points which are the *stereographic projection* of the planes of the upper half of the crystal. This is shown in perspective as the stippled plane of the second diagram and, undistorted, as the finished stereographic projection in the third diagram. Comparing this with the first diagram, note that it is as though you were looking down on the top face of the crystal whose pole comes straight up at you in the center of the projection. The faces parallel to your line of sight have poles on the outer edge of the projection and the sloping faces have poles part way in, whose angular measurement will be discussed later. Figure 2 is a stereographic projection including many more planes of a cubic crystal.

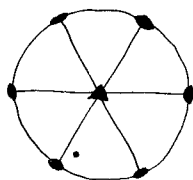
Now consider the circles that outline the sphere in the first diagram. They are drawn solid on the near side, dashed on the far side. Each defines a symmetry plane of the crystal. These have been transferred to the plane of the stereographic projection by connecting every point along the line with the "bottom point" of the sphere as before. Examine the resulting lines in the finished stereographic projection. Notice that the straight lines are the projection of the "vertical planes," those planes that meet in the pole of the (001) face. The "horizontal" plane of symmetry, normal to your line of sight, forms the circular boundary of the projection. The "45° planes" form arcs of great circles through the $\{110\}$ planes.

These are the planes shown in the stereographic projection of point group $O_h - m\bar{3}m$ on the §4 chart, which also shows the poles of a general form, (hkl) (§5), such as $\{123\}$. The planes in the upper half of the crystal are projected as dots. Those in the lower half are projected upward (by inverting the process just described) as circles. Here the two coincide because the crystal has a horizontal plane of symmetry.

USES OF THE STEREOGRAPHIC PROJECTION

A. "Qualitative" Uses, Not Involving Angle Measurement

1. To facilitate consideration of symmetry and the determination of the number of equivalent planes in a form (§5): Consider the point group D_3-32 on the §4 chart. It consists of a 3-fold axis of symmetry with three 2-fold axes normal to it and 120° to each other.



Roughly sketch the stereographic projection of these symmetry elements. Then place a point in a general position (not exactly on or symmetrically between any axes of your figure or on its perimeter) to indicate the projection of the plane (hkl) . Now "act" on this point with the 2-fold axis nearest to it. Remember that it represents a plane normal

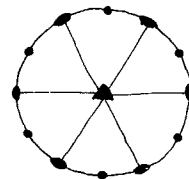
coming up toward you at a small angle to the paper. The 2-fold axis lies in the paper. Mentally keeping one end of the normal at the center of the sphere, swing the other end through 180° around the 2-fold axis. It will then lie in the opposite hemisphere, below the paper, and be represented therefore by an open circle.



Now act on either one of these poles with some other 2-fold axis. Proceeding with the 2-fold axes alone, all the positions shown on the §4 chart for point group D_3-32 will result and they will also obey a 3-fold axis of symmetry normal to the paper.

Now consider a $(10\bar{1}0)$ plane in a crystal of this class. Its normal lies in the plane of the paper, halfway between two crystallographic axes. If its space group is of the type 321 (See Note, §4) this means its normal lies halfway between two 2-fold symmetry axes. If you act on this with the symmetry elements of this point group you will find that this form has only three equivalent planes, not six as does the general form.

There are six points around the perimeter of the projection, but diametrically opposite points on this circle just represent opposite ends of the normal to a single plane.



Notice that the stereographic projection of the general form in D_3-32 on the §4 chart shows you that this point group does not have a *center of symmetry*. If it had, then for every dot there would be a circle on the same diameter of the projection, an equal distance away from the center on the other side, representing a plane in the

other hemisphere related to the first by a center of symmetry, the "opposite end" of the same normal. Compare this with the projections of the general form in classes that do have a center of symmetry.

B. "Quantitative" Use, Involving Angle Measurement

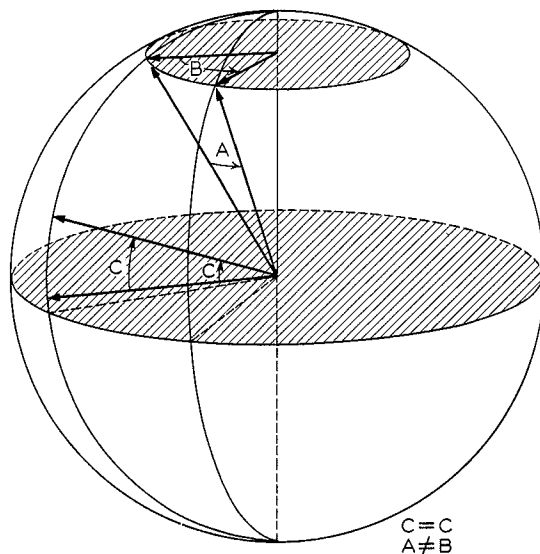
1. *The stereographic net*: From Fig. 1 it is apparent that the relative positions of poles of planes on the stereographic projection are determined by the angles between the planes. It should therefore be possible to construct a grid or network of lines that would enable us to measure these angular relations graphically. Figure 3 is such a net. Both it and Fig. 2 have been left blank on the back so that you may superpose the one over the other in making measurements. There are several ways to use this net.

2. "*Longitude and latitude*" of a pole: Look at the net as though it were the Earth. The *meridians* of longitude run up and down the page; the *parallels* of latitude, from left to right. Superpose Fig. 2 and the position of each pole can be described by its longitude and latitude. This is especially useful in conjunction with the Greninger net. Notice that the meridians, as on the Earth, are *great circles*: In terms of our original sphere (Fig. 1) each one represents the trace on the sphere of a plane passing through the center of the sphere and the poles, i.e., through the axis of the sphere. The parallels are not great circles, except for the equator. They represent the traces on the sphere of planes normal to the axis of the sphere.

3. *Measurement of angles between planes*: On the stereographic net, along each meridian there are 90 divisions, representing 2° each. Along each parallel there are also 90 divisions, representing 2° each. *Only the former may be used for measuring angles between planes.* The reason for this is shown in the accompanying figure. If two poles lie on a meridian *the angle C measured along that meridian is the angle C between the two plane normals.* If two poles lie on a parallel, *the angle B measured along that parallel is not the angle A between the two plane normals.*

To find the angle between any two planes on the stereographic projection, Fig. 1, we must be able to rotate the projection relative to the net so that the two planes in question lie on the same meridian. Then we can read, along the meridian, the number of degrees between them.

Since the *center of the net must coincide with the center of the projection* for this, it may be useful to mount the net on a thick sheet of clear plastic, taking care not to distort it and erect a short needle at its center on which to impale the projection of cubic planes.



E. Leitz, 468 Park Avenue South, New York 16, New York, sells a stereographic net with a device for mounting a tracing sheet over it on a rotatable ring surrounding the net and concentric with it.

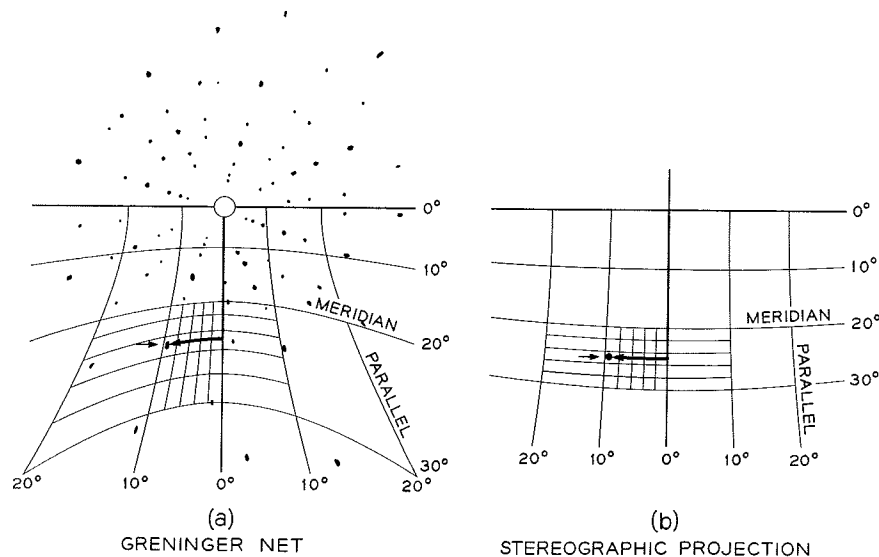
Good clear plastic transparencies of the stereographic net and projections of cubic planes are available from N. P. Nies, 969 Skyline Drive, Laguna Beach, California. Other sources are listed in P. Terpstra and L. W. Codd, *Crystallogometry* (1961). It is difficult to reproduce such a net on paper without distortion. The University of Toronto Press sells a stereographic net of 10-cm radius which is printed on a good grade of paper and appears to be free of distortion.

4. *Use with the Greninger Net:* Since all poles lying on a given meridian represent normals lying in a plane, they represent a "zone," i.e., they are normals from a group of crystallographic planes which are all parallel to a given line. These will give an arc of reflections on a Laue photograph which will lie along or parallel to a meridian line of the Greninger net. This line is divided into 2° segments for reading the angles between the spots. Those arcs transverse to the meridians on the Greninger net are parallels, and readings along these do not give true angles between plane normals. (See paragraph 3, above.)

With a tracing-paper impaled on the needle in the center of your stereographic projection, draw the perimeter circle and mark one point on the perimeter for reference. With a row of reflections along a meridian of the

Greninger net (and the center always at the center of the film) you can measure along the "equator" to the arc of reflections in question and then along the Greninger-net meridian that coincides with this arc, recording the points on the tracing paper with the aid of the stereographic net.

When one arc is finished, rotate the Greninger net about its center until another arc lies on a meridian. The paper on the stereographic projection must



of course be rotated by the same amount before the next zone is plotted. The result will be a stereographic projection of the planes of the photographed crystal. One reason for making such a projection is to check the symmetry around some reflection on the film which was so far from the center that the distortion made the symmetry difficult to detect.

While the symmetry may be more noticeable after the projection, if the point in question was very far from the center it may still be desirable to shift the picture so that the point is centered in the projection. This is very easily done with the stereographic projection as described below.

5. *Shifting the picture:* Referring to the first figure in this section, consider what will happen if the group of normals shown there is rotated *as a group* around the north-south polar axis of the sphere. Their angular relations to each other remain unchanged. In other words, if each pole is shifted *along a parallel of latitude* by the same amount (as measured in degrees *along that parallel*) their angular relations remain unchanged.

Place the point which is to be the center point (after shifting) on the equator. (Remember always to keep the projection center over the net center.) Mark a new point at the center. If this is N degrees along the equator from the original point, then (without shifting the paper in any way relative to the net) mark a new point for each old point, in such a way that every new point is N degrees away from its corresponding old point *along* the parallel on which it lies. In other words, shift each point N degrees along its own parallel.

Your pattern is now centered about the point in question as though you were looking down the normal to that plane.

6. *Stereographic projections of noncubic crystals*: In noncubic crystals the relative dimensions of the unit cell are not the same for one substance as for another. Therefore the angles between planes with given indices are not the same from one substance to another. For this reason, a stereographic projection of the planes of one substance would be useless for any other unless the two substances happened to have the same axial ratios. Stereographic projections of noncubic substances are therefore not often made, but in special cases it may be worth the effort. In one such case a stereographic projection was made of the planes of Scheelite (CaWO_4) which is tetragonal with $c/a = 2.169$. (K. Nassau, *Trans. M. S. AIME* 218: 959, 1960.)

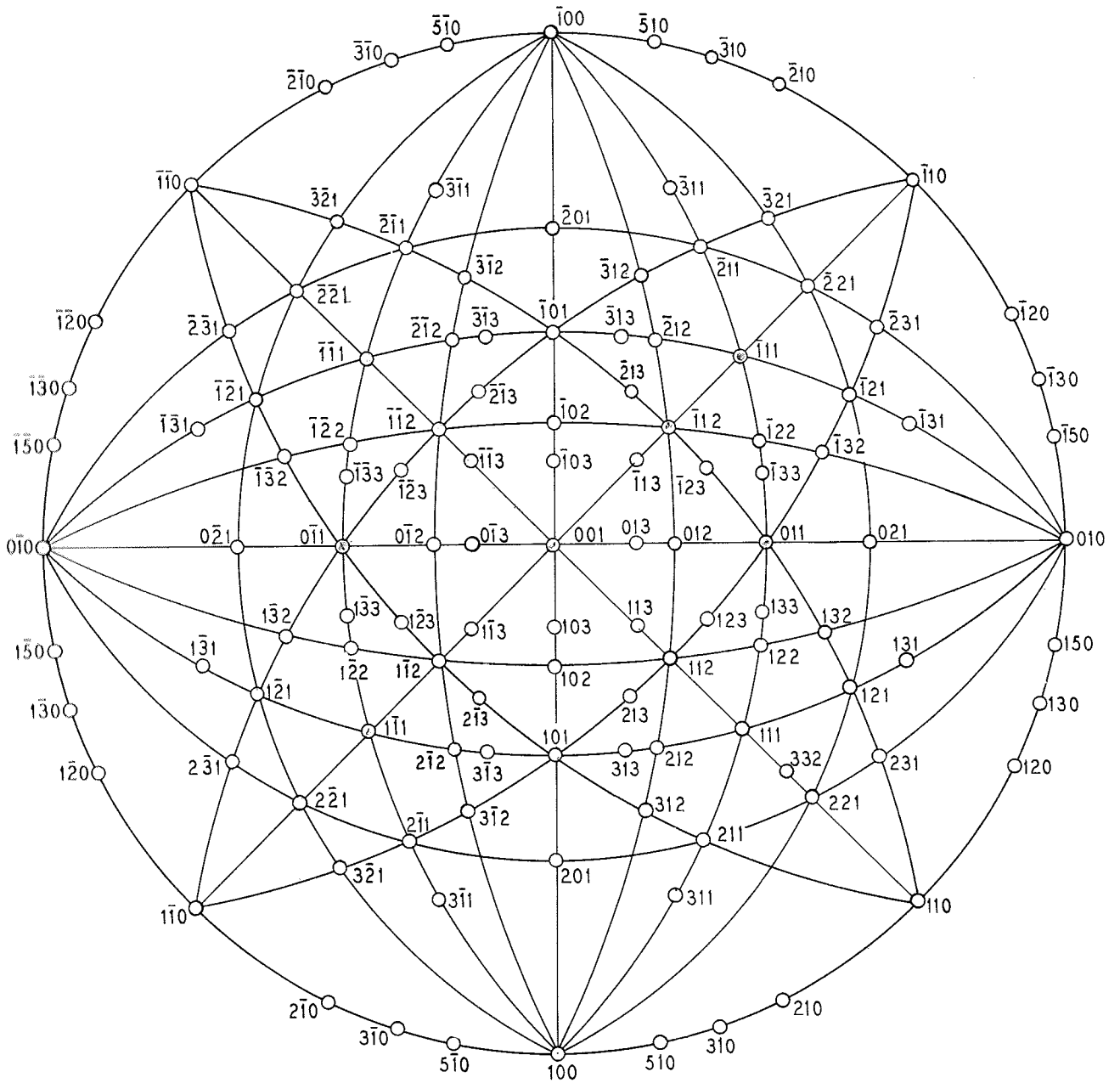


Fig. 2. Stereographic projection of planes in the cubic system onto (001)

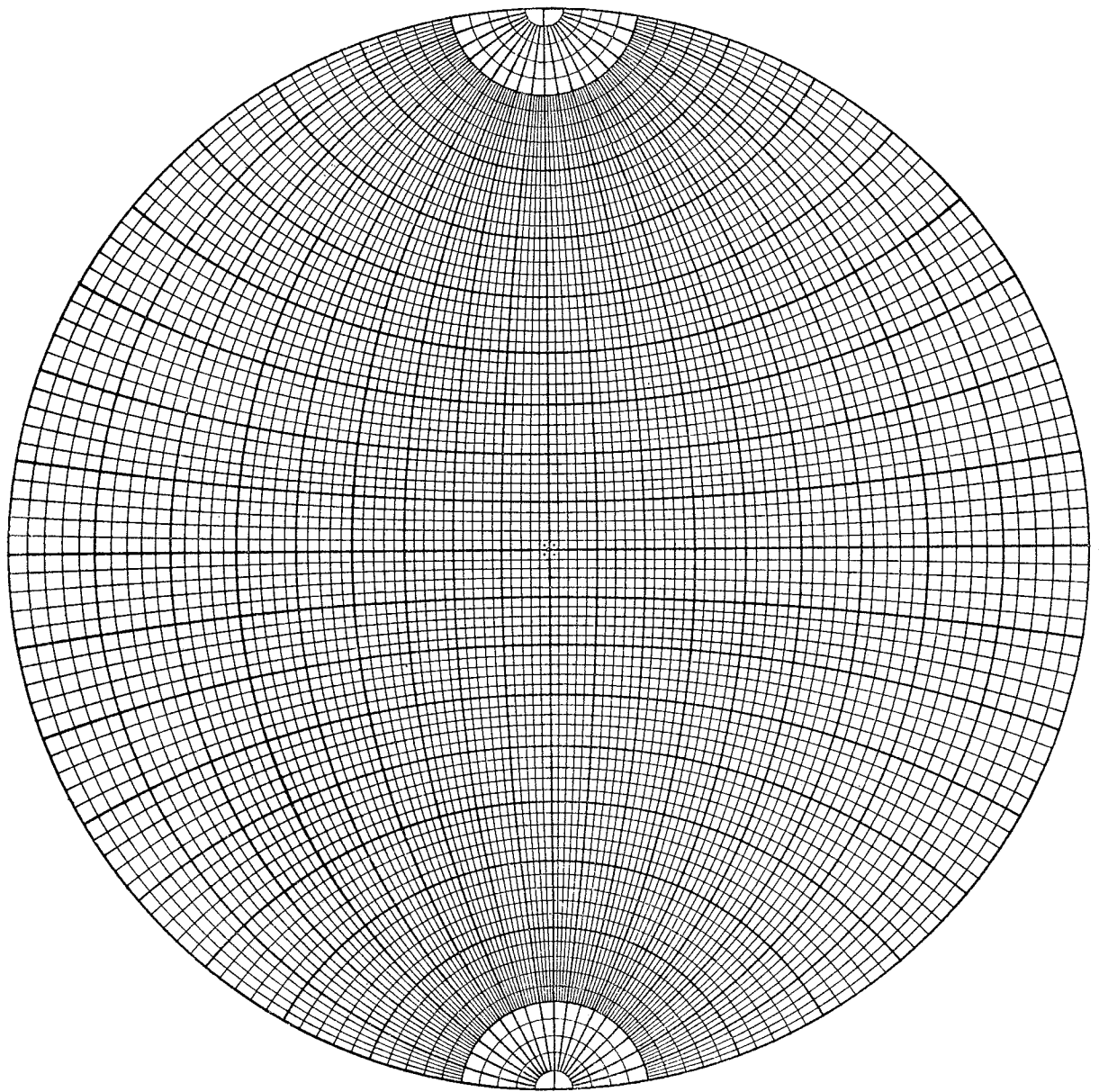


Fig. 3. Stereographic net

Appendix I. The Use of Polarized Light As an Aid in Orienting Transparent Crystals

Useful preliminary information and, in some cases, the complete orientation may be obtained from observation of a crystal by transmitted light with the crystal between crossed polarizers (Polaroids or Nicol prisms with their polarization directions normal to each other and to the direction of propagation of the light). Texts dealing with optical crystallography are listed at the end of this appendix and should be consulted for a fuller understanding of the phenomena described below.

CUBIC CRYSTALS

Between crossed polarizers unstrained cubic crystals appear dark in any orientation, i.e., they are optically isotropic. If a cubic crystal is inhomogeneously strained, it may transmit light, but the light-transmitting regions will shift gradually across the crystal as it is rotated (around the light path) between crossed polarizers. This phenomenon is readily distinguishable from those described below.

NONCUBIC CRYSTALS

All other crystals are optically anisotropic and will appear bright between crossed polarizers, except in certain positions. When any anisotropic crystal is rotated around the line of sight (light path) it becomes dark every 90° unless the light path is along an optic axis. All noncubic crystals have either one optic axis ("uniaxial") or two optic axes ("biaxial") according to their symmetry.

Uniaxial crystals (tetragonal, hexagonal, trigonal): The optic axis lies along the axis of highest symmetry (which is always chosen as the c crystallographic axis).

If this axis is parallel to the light path and sight path, rotation of the crystal around this axis will cause no change in its light transmission. If it is thin, it will appear continuously dark.

If the optic axis is not parallel to the light path and sight path, then as the crystal is rotated around this path it will become dark whenever the optic axis lies in the plane defined by the sight path and the polarization direction of either polarizer. This will happen every 90° of rotation. (See Fig. 1.)

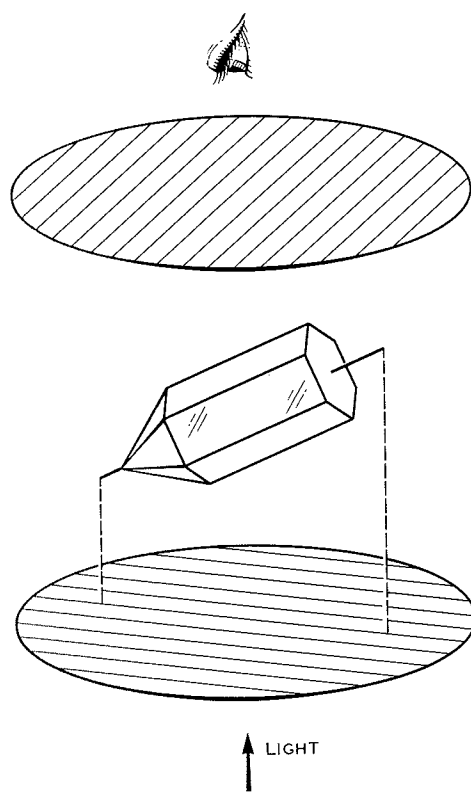


Fig. 1

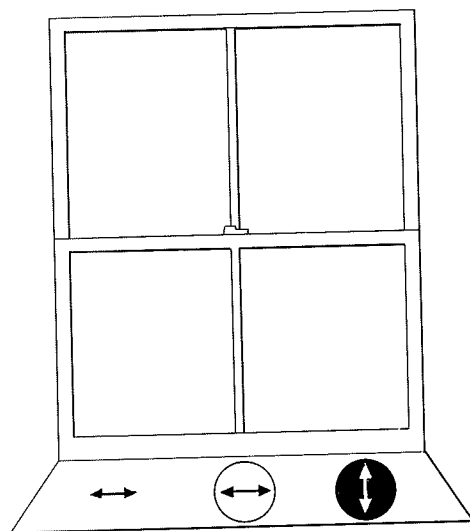


Fig. 2

A knowledge of the polarization directions of your polarizers (and a little three-dimensional thinking) will enable you to bring the optic axis of your crystal into parallelism with the light path and sight path. The polarization direction of a polarizer may be quickly determined approximately by looking through it at a flat, brightly reflecting surface such as an enameled window sill or a glass microscope slide. Light from such a surface is largely polarized parallel to the reflecting surface, and of course normal to the direction of propagation of the light. The surface will therefore look light when viewed with the polarization direction of the polarizer parallel to the surface, and dark when the polarizer has been turned 90° . (See Fig. 2.) In most polarizing microscopes, the polarizer (below the stage) is usually kept with its polarization direction from front to back in the field of view and the analyzer (above the stage) with its polarization direction from left to right in the field of view.

Since the optical symmetry of a uniaxial crystal is that of an ellipsoid of revolution, i.e., ∞/m , the optical properties can give you no information about azimuth angle around the optic axis.

Biaxial crystals (orthorhombic, monoclinic, triclinic): In optically biaxial crystals, the two optic axes, which make an angle with each other characteristic of each different substance, define a plane known as the *optic plane*. Unless the optic axes are at 90° to each other (a rare accident, which would be true only at one temperature and wavelength of light, since the axial angle varies with both these parameters), there is an *acute bisectrix* (Bx_A) and an *obtuse bisectrix* (Bx_O) lying in the *optic plane*, and an *optic normal* normal to the *optic plane* (Fig. 3).

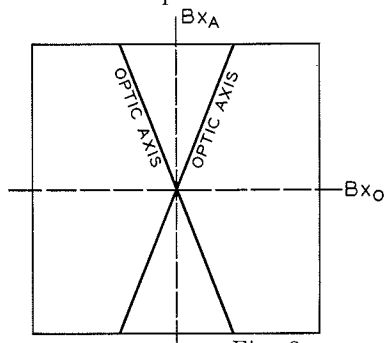


Fig. 3

These three directions are thus normal to each other and each is a 2-fold axis of symmetry of the optic plane with its axes. Consideration of the symmetry of the three optically biaxial systems, orthorhombic, monoclinic, and triclinic (§4), will show how the optic plane must be oriented in each.

In the *orthorhombic system* any property of a crystal must have a 2-fold axis of symmetry parallel to each of the three crystal axes, or a 2-fold axis parallel to one, and planes parallel to that and the other two. Consideration of the symmetry of the optic plane will show that the three directions, the Bx_A , Bx_O , and optic normal must lie parallel to the three crystallographic axes in the orthorhombic system, but any one of these directions may lie along any one of the crystallographic axes.

Similarly, in the *monoclinic system*, one of the three directions, the Bx_A , Bx_O , or optic normal, must lie along the b axis (the 2-fold axis or the normal to the symmetry plane, see §4), but the other two may have any orientation in the plane normal to b (remaining, of course, normal to each other).

In the *triclinic system* there is no restriction whatever on the orientation of the optic plane.

EXTINCTION POSITIONS

The extinction position of a crystal is that position during rotation between crossed polarizers at which it appears dark. Consider now the three directions, the acute bisectrix (Bx_A), the obtuse bisectrix (Bx_O), and the optic normal—whenever the sight and light path is along one of these three, the crystal will be at the extinction position when the other two are parallel to the polarization directions of the polarizers. Note that whereas the extinction position of uniaxial crystals occurred only when the optic axis lay in the polarization plane, this is not so in the case of biaxial crystals.

TEST FOR AN UNSTRAINED SINGLE CRYSTAL

In an unstrained single crystal, extinction will occur simultaneously throughout the crystal as it is rotated between crossed polarizers. If it is inhomogeneously strained, the dark regions of the crystal will shift across the crystal continuously as the crystal is rotated. If the specimen is not a single crystal, there will be sharp boundaries between parts that are at extinction (dark) and parts that are not (light). If the crystal is twinned, the two parts of the twin may or may not extinguish differently. An example in which they do not is provided by the trigonal crystal quartz (SiO_2). In one type of twinning in quartz, the parts of the twin are related by a rotation of 180° around the c axis. Since this is also the optic axis (see above), the appearance of the crystal in cross-polarized light is not affected by this twinning.

Sharply bounded regions which show different color between crossed polarizers but the same extinction position may indicate twinning, but more commonly are due to abrupt changes in thickness due to surface steps.

RECOMMENDED TEXTS

- Bunn, C. W. *Chemical Crystallography*. 2d ed. Oxford, Clarendon Press, 1961. Chapters 3 and 4.
- Kerr, Paul F. *Optical Mineralogy*. New York, McGraw-Hill Book Company, 1959.
- Wahlstrom, E. E. *Optical Crystallography*. New York, John Wiley & Sons, 1943.
- Winchell, A. N. *Elements of Optical Mineralogy*. Part I. New York, John Wiley & Sons, 1937.

Appendix II. Examples of Laue Photography

Sample orientation and Laue photography by Paul H. Schmidt.

Samples of Laue photographs of several simple substances of known orientation are presented here to aid the reader in the recognition of crystal symmetry in Laue photographs.

Note that the curves passing through all spots of a given "zone" (§18) meet at a few well-defined points which are reflections from important planes in the crystal, in some cases planes normal to symmetry axes.

If the beam encounters two crystals, their superposed patterns will give a complex array of spots with many intersecting rows. If the two are twin-related, the two sets of spots will be symmetrically related to each other. A fine polycrystalline specimen gives a pattern of concentric circles of very small spots. With increasing grain size there is every gradation between this and the single-crystal patterns.

If the crystal is inhomogeneously strained, the spots will be streaked or distorted and each spot may show a pattern of light and dark which is related to the distribution of strain in the small region irradiated by the beam.

All photographs were taken with tungsten radiation, except as otherwise stated, and at a specimen-to-film distance of 5 cm, except as otherwise stated. The original size of the photographs, before reduction, was $4\frac{1}{4} \times 6\frac{1}{2}$ in.

The caption for each single-crystal photograph gives the direction in the crystal that was approximately parallel to the beam. On most photographs a known direction $[uvw]$ normal to the beam is also indicated.

LAUE PATTERNS OF FACE-CENTERED AND
BODY-CENTERED CUBIC CRYSTALS

In face-centered cubic crystals, reflection will only occur when h , k , and l are all odd or all even (with zero considered as an even number). See §23. Thus, the first-order reflection from the (103) plane, for example, cannot occur. Laue spots that occur in the position for the (103) planes will be due to second-order reflection (with higher even-order reflections superimposed, resulting from different wavelengths; see §10 and the first paragraph of §18). Such a spot is labeled (206) in the diagram below, to call attention to this fact, but in the photographs on the following pages the order of the reflection will not be indicated. In body-centered cubic crystals, reflection will only occur when the sum of the Miller indices is even.

The center regions of the Laue patterns of a body-centered cubic crystal and a face-centered cubic crystal are shown here. Note that because of the restrictions mentioned above, the zones made up of $\{h0l\}$ reflections, such as (103) , extend farthest toward the center of the pattern in the body-centered case, while the zones made up of $\{hhl\}$ reflections, such as (114) , extend farthest toward the center of the pattern in the face-centered case. Compare Fig. 1 with Fig. 2 on the following page.

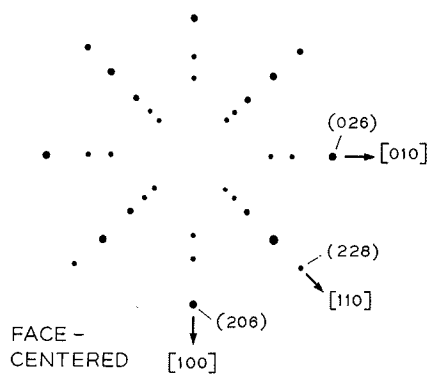
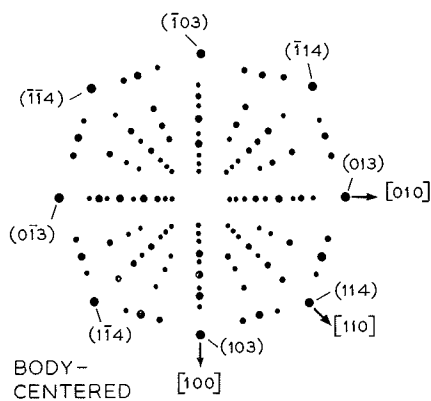
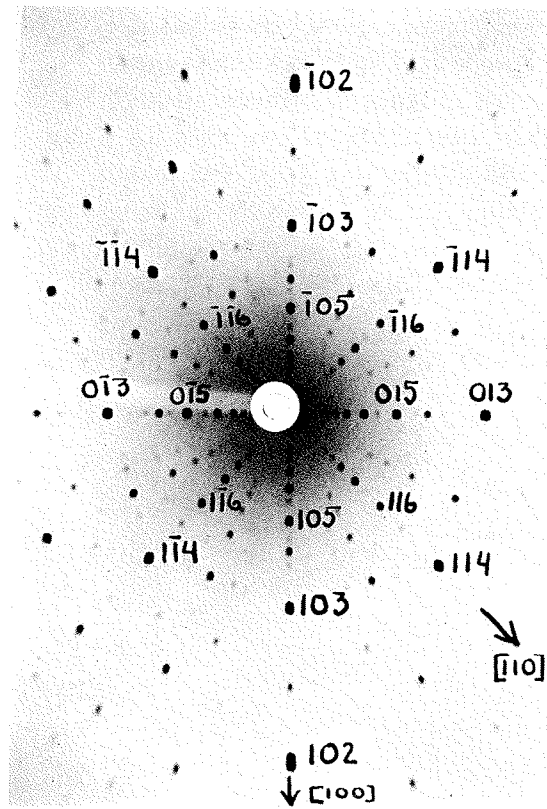


Fig. 1. Face-centered cubic crystal (aluminum) (cubic close-packed)
 Beam approximately parallel to [001].



[66]

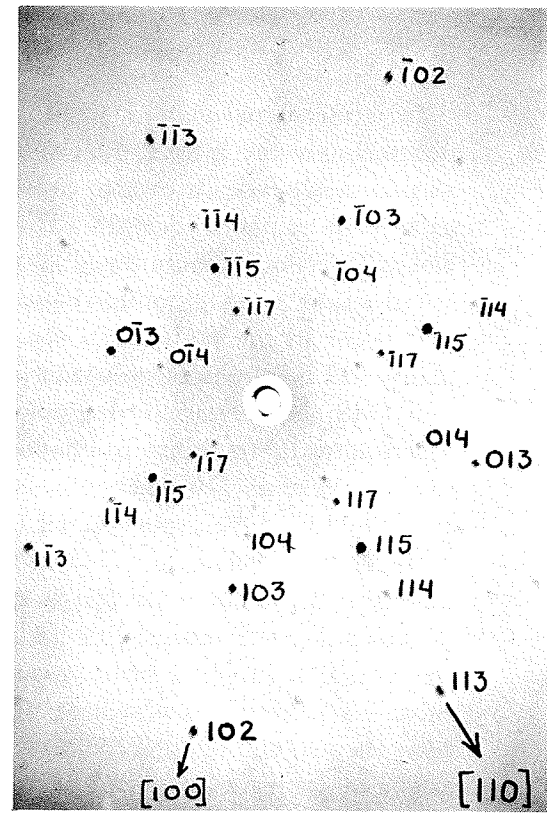


Fig. 2. Body-centered cubic crystal (tungsten)
 Beam approximately parallel to [001].

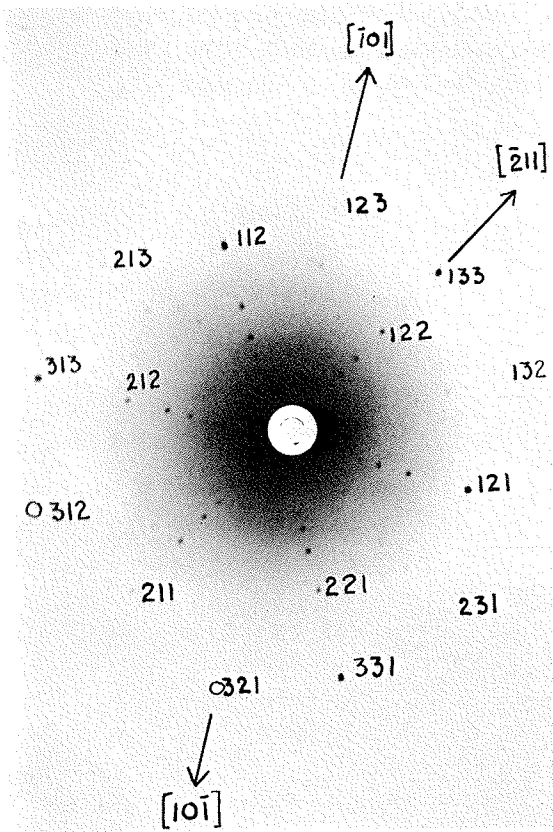


Fig. 3. Face-centered cubic crystal (copper)
Beam approximately parallel to $[111]$. Compare Fig. 4.

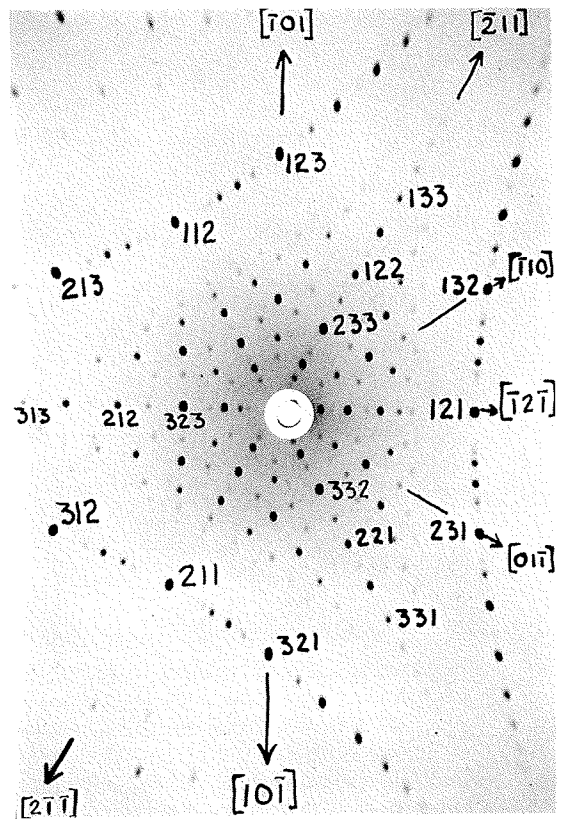


Fig. 4. Body-centered cubic crystal (tungsten)
Beam approximately parallel to $[111]$. The $[011]$ direction is $35^{\circ}16'$ from $[111]$ toward $[\bar{2}11]$. Compare Fig. 4a, §25.

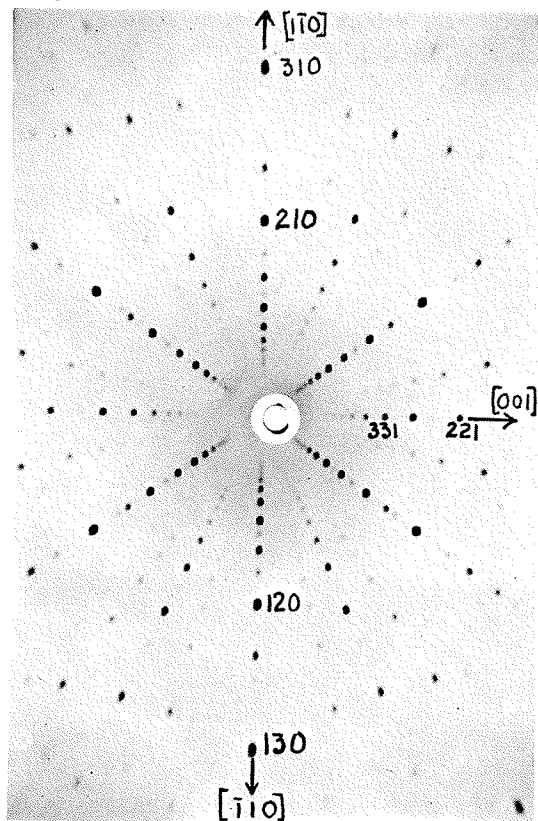


Fig. 5. *Body-centered cubic crystal (tungsten)*
 Beam approximately parallel to $[110]$. The $[100]$ direction is 45° from $[110]$ toward $[\bar{1}10]$.

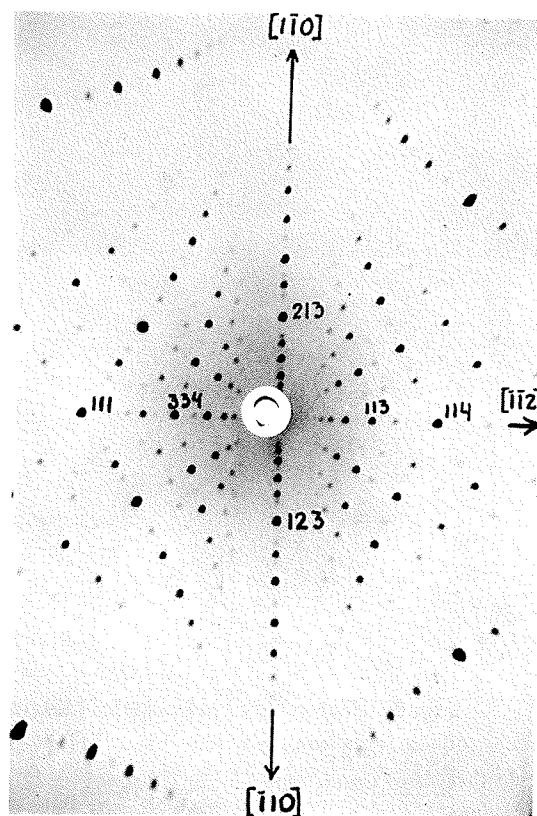


Fig. 6. *Body-centered cubic crystal (tungsten)*

Beam approximately parallel to $[112]$. This pattern does not have a two-fold symmetry axis normal to the film although at first glance it appears to have. The $[011]$ direction is 30° from $[112]$ toward $[\bar{1}10]$. The $[001]$ direction is $35^\circ 16'$ from $[112]$ toward $[\bar{1}12]$.

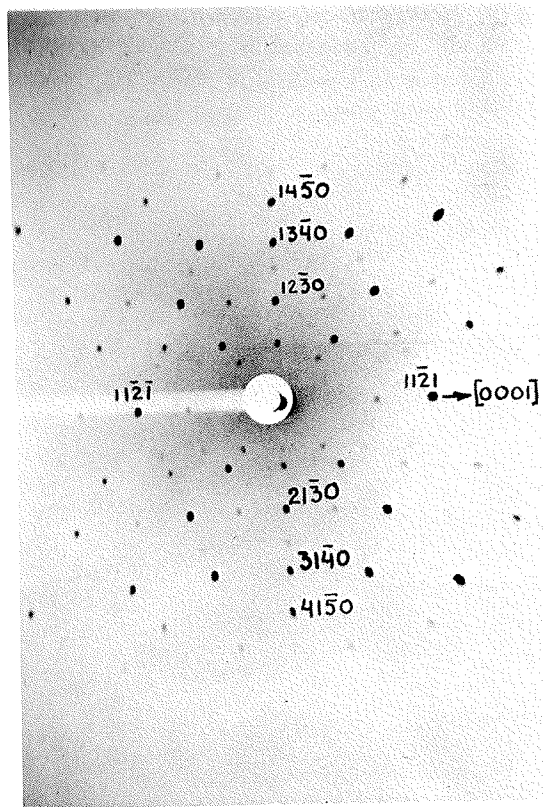


Fig. 7. Hexagonal close-packed crystal (cadmium)

Beam approximately parallel to $[11\bar{2}0]$, i.e., parallel to the a axis.

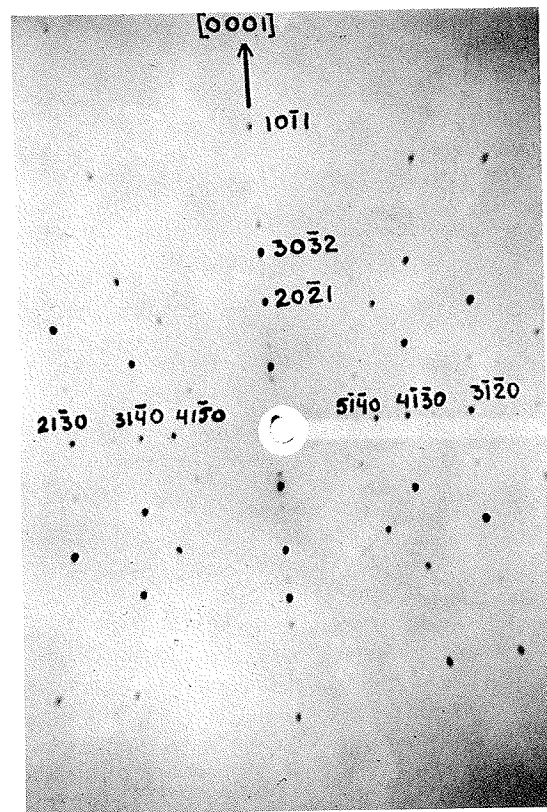


Fig. 8. Hexagonal close-packed crystal (cadmium)

Beam approximately parallel to $[10\bar{1}0]$.

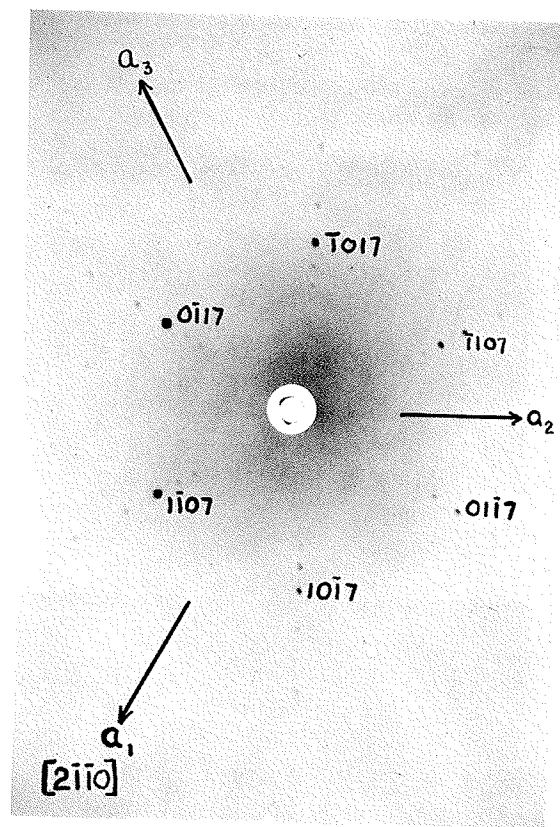


Fig. 9. Hexagonal close-packed crystal
(cadmium)

Beam approximately parallel to $[0001]$, i.e., parallel to the c axis.

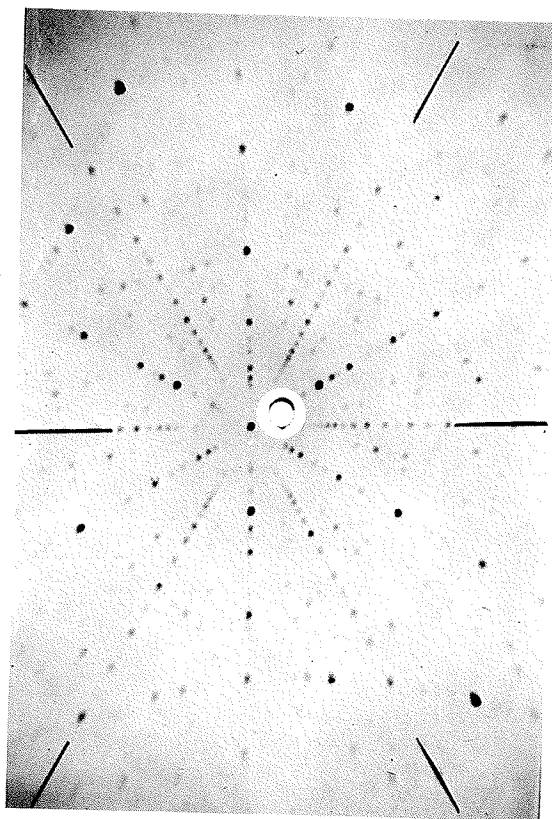


Fig. 10. Trigonal crystal
(corundum, Al_2O_3 , "sapphire")

Beam approximately parallel to $[0001]$. The a axes, $\langle 11\bar{2}0 \rangle$, lie in the directions indicated by the short black lines. There are 2-fold symmetry axes parallel to them and mirror planes normal to them.

[70]

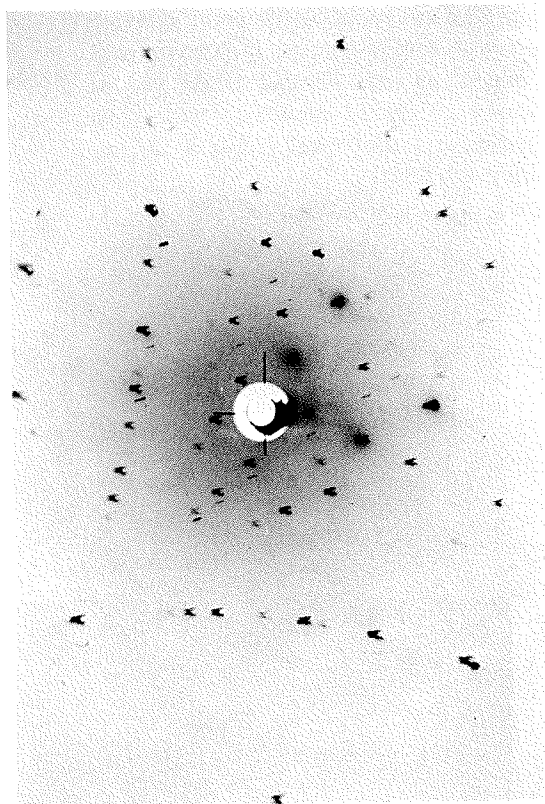


Fig. 11. Distorted spots indicating inhomogeneous strain (bismuth)

In a soft substance such distortion is readily produced. If the damage is only surface damage it may be removable by etching (i.e., by chemically removing the surface layers).

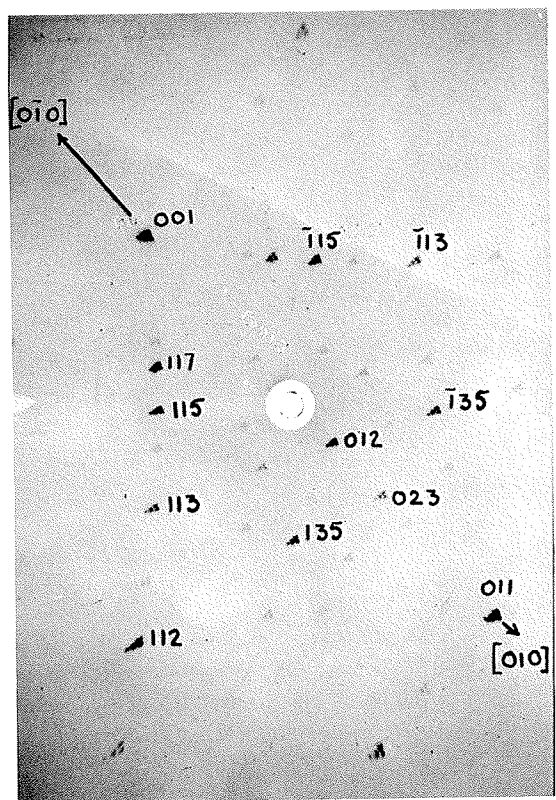


Fig. 12

Each spot is composed of several smaller spots indicating that the area in the beam was composed of several crystallites whose structures were nearly, but not quite, parallel to each other. Such subparallel orientation may occur during growth, when it is called "lineage" (M. J. Buerger, *Z. Krist.* 89: 195, 1934). (Aluminum.)

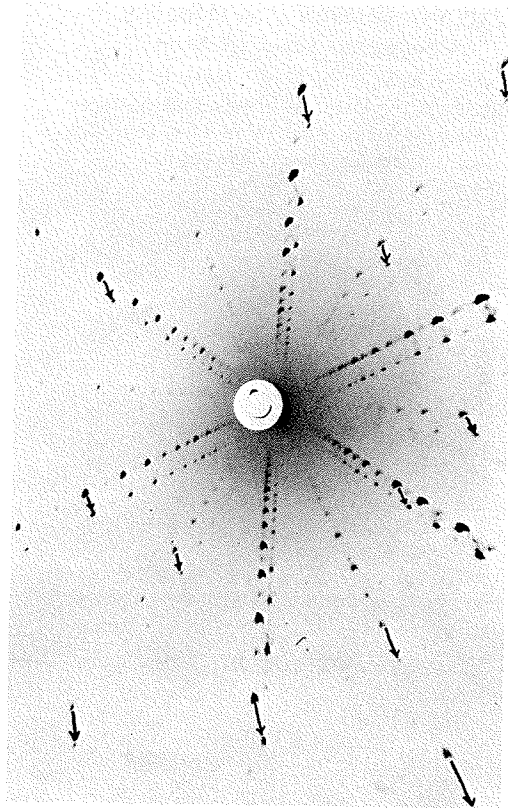


Fig. 13. Two crystals in the beam, differing in orientation by a rotation of approximately 2° around an axis parallel to the film as indicated by arrows connecting spots from equivalent crystallographic planes in the two crystals (rhenium)

Beam approximately along $[0001]$.

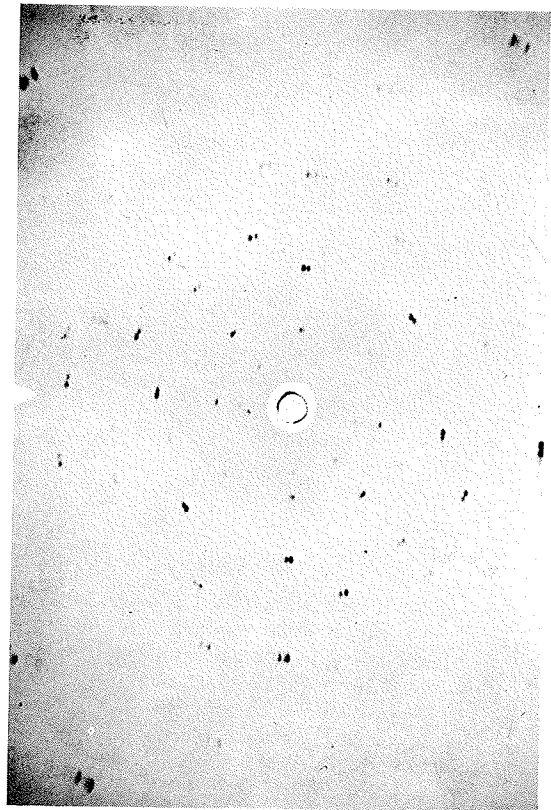


Fig. 14. Two crystals in the beam, differing in orientation by a rotation of approximately 2° around an axis normal to the film as indicated by arrows connecting spots from equivalent crystallographic planes in the two crystals (cadmium)

Beam approximately along $[11\bar{2}0]$.

[72]

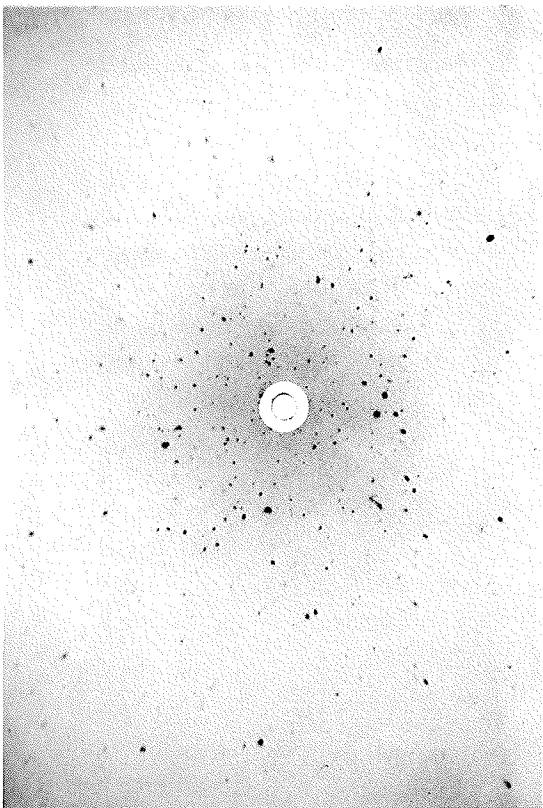


Fig. 15. Several crystallites in the beam

A coarsely polycrystalline specimen gives such a picture. A roughly abraided or sawed surface of a single crystal could also give such a picture.

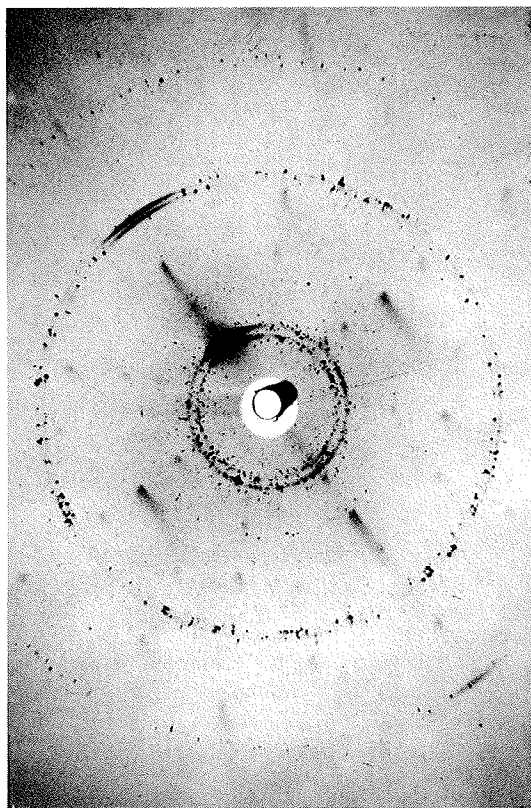


Fig. 16. An aluminum crystal with an abraided surface, taken with copper radiation

Of the many randomly oriented crystallites on the surface only those which are oriented so that they satisfy the Bragg equation for the characteristic radiation (§17) will give strong enough reflections to show on the film. These reflections show as rings of a given θ . Reading from inside outward, the rings on this film result from: $\text{CuK}\alpha_2$ radiation from the $\{511\}$ planes, $\text{CuK}\alpha_1$ from the $\{511\}$ planes, $\text{CuK}\alpha_2$ from the $\{422\}$ planes (i.e., second-order reflection from $\{211\}$), $\text{CuK}\alpha_1$ from the $\{422\}$ planes, and $\text{CuK}\beta$ from the $\{511\}$ planes. There are also spots from the planes of the underlying single crystal which are reflecting rays of various wavelengths from the white radiation (§17).

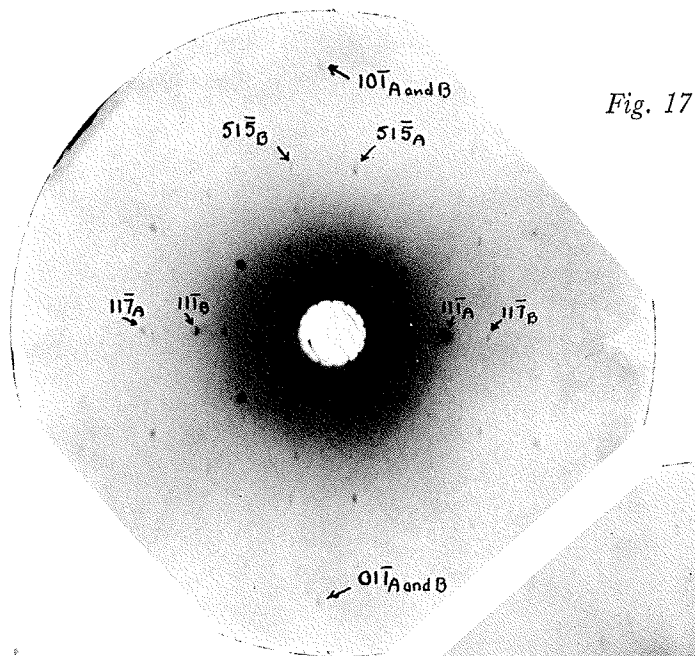


Fig. 17

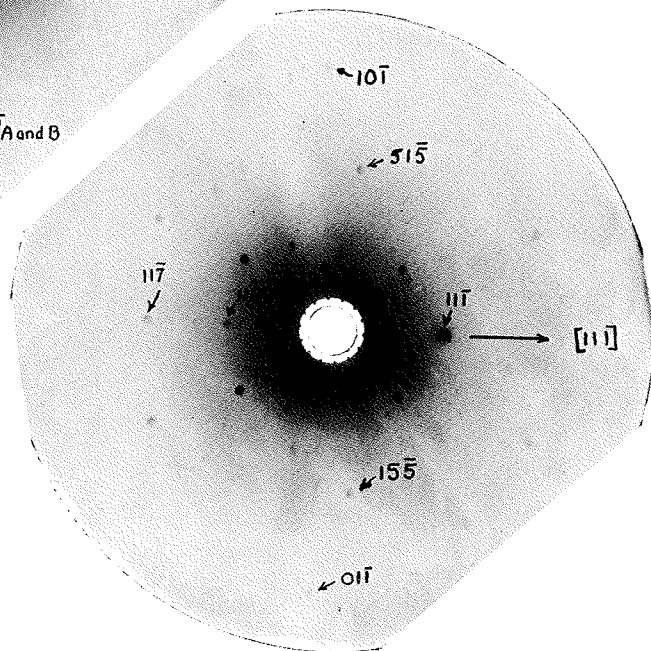


Fig. 18

Figs. 17 and 18. Laue photographs of a silicon crystal with the beam hitting the crystal at the composition plane of a twin (Fig. 17) and with the beam hitting the crystal near but not at the composition plane (Fig. 18) (see *Twinning* §13)

The twin plane (and composition plane) is $(11\bar{1})$. This plane is normal to the film and vertical in Fig. 17. It thus results in a mirror plane passing vertically through the center of the pattern. Half-spots on one side of this plane, such as $(11\bar{1})_A$, $(5\bar{1}5)_A$, and $(11\bar{7})_A$ are mirrored on the other side of this plane as $(11\bar{1})_B$, $(5\bar{1}5)_B$, and $(11\bar{7})_B$. More than half of the beam was hitting the A part of the crystal. The A spots are therefore larger than the B spots. Since the (101) spot and the $(01\bar{1})$ spot lie on the mirror plane, they are unaffected by the twinning. (Copper radiation, specimen-to-film distance 3 cm.) Crystal selected and photographed by D. F. Gibbons.

Appendix III. Rhombohedral Crystals

Crystals in which a rhombohedron is the shape of the smallest unit of structure (the "unit cell," see §3) are called *rhombohedral* and their space-group symbol (§11) begins with the letter *R*. Their unique axis of symmetry is 3-fold, i.e., they are trigonal. The word was formerly used to refer to all trigonal crystals, but it is now known that many trigonal crystals have a unit cell which is the shape of the primitive hexagonal cell. Their space-group symbol begins with *P*.

The rhombohedron is like a cube which has been extended or shortened along the cube diagonal, [111]. Its shape is thus completely defined by the length of the edge a and the angle α between two edges which meet in the 3-fold axis.

Miller indices of faces and directions of rhombohedral crystals can be expressed on hexagonal axes and it is generally more convenient to use this system of axes. Formulas for conversion from one system to the other are given below.¹

Given rhombohedral, to find hexagonal: Lattice constants

$$a_H = 2a_R \sin \frac{\alpha}{2}$$

$$c_H^2 = 9a_R^2 - 3a_H^2$$

Indices

$$h_H = h_R - l_R$$

$$k_H = k_R - h_R$$

$$i_H = -(h_H + k_H)$$

$$l_H = h_R + k_R + l_R$$

Given hexagonal, to find rhombohedral: Lattice constants

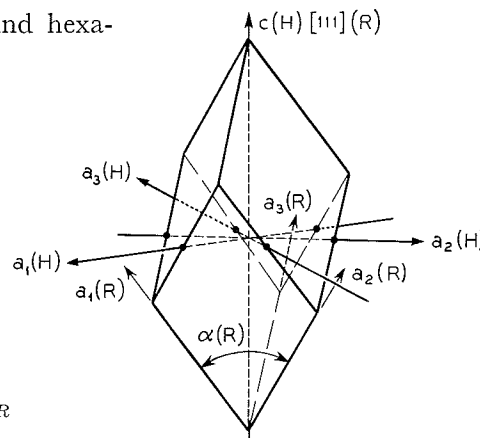
$$a_R^2 = \frac{a_H^2}{3} + \frac{c_H^2}{9}$$

Indices $3h_R = h_H - k_H + l_H$

$$3k_R = h_H + 2k_H + l_H$$

$$3l_R = -2h_H - k_H + l_H$$

$$\sin \frac{\alpha}{2} = \frac{3}{2 \left[3 + \left(\frac{c_H}{a_H} \right)^2 \right]^{1/2}}$$



¹ See the *International Tables for Crystal Structure Determination* for a fuller discussion of hexagonal-rhombohedral relationships.