July 30, 2000

Experiment X3

Be sure to read the notes "Exposure to Ionizing Radiation" before starting. Nick will check you out on the use of the x-ray equipment prior to use. You are responsible for monitoring the radiation to make sure you have proper shielding and for filling out the log each time you use the x-rays. If you have no previous experience with x-rays, you might also read the notes for experiment X-1 for introductory references on x-ray production, absorption and safe dose. Cullity and Warren(see references) also discuss some of the basic material.

Transmission and Back-reflection Laue Photographs

1. Take a Laue transmission photograph of one of the alkali halide single crystals provided using 34 KV molybdenum radiation, 10 ma and 4 cm crystal to film distance. With such a crystal, a principal crystallographic axis is normal to a cleavage plane. Your photograph should be taken with the beam aligned as close as possible to this axis (see remarks on alignment below). You will probably need to take at least two photographs to get the alignment reasonably precise.

   Plot the gnomonic projection of each photograph, using the D= 4 cm projection ruler provided *. Index the spots (Miller indices), and use the short wave length limit, the intensity information and the missing spots to estimate the upper and lower bounds on the lattice constant, assuming the crystal is cubic. Show that the symmetry of the picture agrees with the assumption the crystal is in fact a cubic one †. From the missing spots, is the crystal fcc or bcc? What are the possible alkali halides?

2. Take a back-reflection Laue photograph of a silicon single crystal with a film to crystal distance of 3 cm. Use the Greninger chart provided and plot a stereographic projection of the principal reflections. From this projection, find the orientation of the vertical (i.e. the line of the x-ray beam through the specimen) and the unit triangle formed by the zones [001],[011],[111]. Identify the other principal zones, and assign Miller indices to the principal spots. This can be done by using the table of angles between crystallographic planes or by rotating the stereographic projection into the standard [100] chart provided (see also Cullity). Finally, give the orientation of the crystal relative to its holder (assumed to be parallel with the film). State your precision.

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* See the notes for how to use the ruler

† Is there any other possibility? See reference 6

References †

*1. B.D. Cullity, *Elements of X-Ray Diffraction*, Addison-Wesley 1956. This book is in the P410/510 library. See especially pages 215-236. Cullity is a good reference on the stereographic projection.

*2. B.E. Warren, *X-ray Diffraction*, Addison-Wesley 1969. This is in the Lab library. There is also a Dover reprint in 1990 which is available at low cost from Amazon.com. There is an introduction to basic x-ray diffraction theory, and a chapter on the Laue method.

*3. Henry, Lipson and Wooster, *The Interpretation of X-ray Diffraction Photographs*, Macmillan 1960, 2cnd edition, pages 34-41 and 71-80.

*4. Special Notes by L.G.Parratt (1955)

5. Barrett and Massalski, *Structure of Metals*, McGraw-Hill 1956, pages 215-229.

6. Amoros, Buerger and Amoros, *The Laue Method*, Academic Press 1975. This is in the Physical Sciences Library: QD945.A52 L3.

7. In a 1989 Cornell Ph.D. thesis Brenda R.S. Temple used the Laue method to analyse the structure factors for complex substances such as lysozyme. There are some beautiful pictures of the Laue patterns, and a detailed account of the analysis. This is much more advanced than what we do in this lab, but it may be worth a look. QC10 19889 T284.

† "*" indicates some xeroxed material included in these notes.

Some perhaps useful notes on the X3 apparatus from a non-expert

In this experiment, both transmission Laue and back-reflection Laue photographs are made. The transmission photograph is made with the X-ray beam normal to the crystal face—a cleavage plane. To set this up, you first align the crystal as well as possible by eye using the goniometer to make the adjustment. Rotation about a vertical axis is usually aligned already, it is only the horizontal axis you need to worry about. Take one exposure for 2 hours. If the crystal is perfectly aligned, the spots will be symmetrically placed around the central beam spot. If it is misaligned, you have a projection onto a tilted plane. It is just a matter of geometry to calculate the additional tilt needed to bring the crystal into alignment. You can use the goniometer scale to make the necessary adjustment. Be sure you keep track of the sign as well as the magnitude of the change, otherwise another picture will be required to correct your mistake. With care, your second picture will show perfectly aligned spots in both directions.

Previously, a 4° misaligned crystal was also photographed. (See the L.G.P. notes) This is no longer necessary. We found the old laser alignment difficult to use, and not really necessary, so have abandoned it.

The film cassette needs to be handled a bit carefully. The front is simply black paper to let X-radiation through to the film set down against it and backed with a plate to hold it flat. Loading is done in the dark. (Ask Nick for instructions about loading and developing. Be sure that the developer and fixer are fresh— we have had trouble and wasted time in the past with outdated developer.) The lower edge of the cassette has a groove in it which fits on a rail to position it 4 cm from the crystal. A little lever at the top edge swings down into a slot to clamp it there. Exposure will be about two hours at 34 KV and 10 ma with the Mo tube. The assembly sits on a three-legged aluminum stand which in turn is placed on a $\frac{1}{4}$" plywood sheet to get it high enough. The X-ray beam comes out of the tube ports inclined downward by about 20° . The spring-loaded shield is pulled down from the port opening and the snout on the front of the collimator is poked into the aperture at the tube window. The three leveling screws in the base of the assembly enable one to make the adjustment. (Again, ask Nick to help with this if necessary.) The back end of the base is practically down on the aluminum support stand. In any event, collimation will be checked after the X-rays are turned on. The main switch on the wall is thrown up; the line button is pressed, energizing the power supply rack. The water circulating pump must be turned on before this is done. (Listen for the "click".) Turn the voltage control to zero, the current control switch to A(which will hold stable a current up to 10 ma; other positions control higher levels) and press the high voltage button. Current should come to 10 ma. Then raise the voltage with the Variac on up to its maximum value of 34 KV. It does not hurt to have the lead shields disposed around the tube windows to intercept any leakage from the tube. Be sure the small lead shield is also in place around the beam itself—this greatly reduces background radiation. To check collimation (the photographic plate is not yet in place) a strip of fluorescent material on a stick is used. Behind the first pin hole a bright spot should show on the strip. If alignment is correct, a spot will also be seen behind the second pin hole; it may be necessary to swing the apparatus back and forth a bit on the table to maximize

this second collimation. A much weakened spot should be visible behind the crystal. Turn off the voltage(it is not a bad idea to turn down the control Variac as well), put the loaded cassette in position, turn the little cup to intercept the direct transmitted beam and then proceed to energize the X-ray tube as before. At the end of the two hour exposure, turn the little interceptor cup out of the beam for a few seconds to index the position of the zero order diffraction in the exposure.

For the back reflection picture, the tube current control is turned to position B, where the current will be held at 15 ma for the 34 KV, one hour exposure. The goniometer arrangement and plate positioning are different than for the transmission pictures. The goniometer was built by Mr. Szabo and because of some uncertainty at the time of construction, it was made for both a 30 mm and a 50 mm plate-to-crystal distance, for each of which distances a Greninger net is available in the laboratory. We generally use the 30 mm net and distance but have not modified the apparatus to make the 50 mm distance unavailable. Just be careful into which the plate holder is put. Two long screws, hand tightened only, are used with the 30 mm position, and two shorter thumb screws for the 50 mm spot. Use the long screws generally, therefore.

Since in back-reflection, the X-rays come through the film to the crystal and then are diffracted back to the film, the film is perforated with a central hole to allow a collimator to be screwed in place after the film is loaded. Again a black paper cover to thecassette, faces the crystal, the back side of the cassette being metal to shield the film from direct radiation from the tube. To load the cassette, the collimator is first unscrewed (threaded end projecting through the cassette away from the crystal) and removed. The several thumb nuts around the rim removed and the aluminum frame and paper cover lifted off. Note two dowel pins at the rim for positioning the frame onto the back. With lights out a film is dropped onto the back centered within the feld seal on the edge. The cover is put on and thumb nuts screwed on. We now have to punch a hole through the film for the collimator. A jig, properly doweled for accurate positioning is laid across the front of the now sealed cassette and the assembly placed on the base of the bench hand press. The jig carries a small punch which matches the central hole of the cassette. This is spring loaded to be in the up position. Travel of no more than a quarter of an inch carries the punch through the film. **Do not lean on the press handle as though your life depended on it.** It would be well to practice on an old film with the lights on to see how it all goes. Take it easy, anyway. After the hole is punched the collimator is put through the hole (threaded end away from the crystal again) and the securing nut screwed on to it. You're now ready to mount things up at the X-ray tube window.

It will be noted that the goniometer and plate mount slides back and forth on the pedestal, collimator end rising as it moves toward the X-ray tube. For ease in alignment and to reduce radiation at the sides, the screwed end of the collimator is fitted with an extension tube which just slides nicely over the threaded end. The long extension is used for the 30 mm plate to crystal distance and the short one for the 50 mm distance. These extensions are in the plastic box on the base of the pedestal. Put on the proper extension, slide the goniometer forward on the pedestal until it is at window height (spring loaded X-ray shield drawn

down) and then move the whole business, pedestal et al, forward until the collimator extension fits inside the recess at the window. Actually, before this is done it is easier if the plate is positioned and clamped first in the proper slot (30 or 50 mm, 30 for most of our work here). The paper side of the cassette faces the crystal. Along one edge of the back metal side of the cassette is a milled recess and two dimples. This edge goes down and the two clamping screws fit into the dimples to hold the film cassette in place. Hand tighten is enough. Now move the whole works with the collimator extension poking into the window recess. Now take your picture. From symmetry, the Greninger net, and the readings on the divided circles, along with a copy of Elizabeth A. Wood's "Crystal Orientation Manual" † you can turn the crystal so as to get what orientation is sought. (Note that the clamped stem of the crystal mount has a circle divided in 60° intervals clamped to the end of the stem where it protrudes from *its* clamp.)

Also be aware that X-ray film is sensitive on both sides(double coated) so you don't have to worry about which side is up in the cassette. Note also: On the developed film, X-ray shadows of four accurately positioned aluminum pointers on the paper front of the cassette, will serve to locate the "center" of the picture.

† This book was lost, so you will have to do without it, but I retained the reference to it here in the original notes.

Using the Special Ruler for obtaining Miller Indices of a Laue Spot

The spot on the film can be converted directly to a graph from which Miller indices can be read off. This is called a "gnomonic projection". The mathematical equations are given in the body of these notes, and in the appendix at the end. There is also an example in the notes. Here we discuss practical use of the special ruler, which is a brass strip 1 1/4" wide by 35 1/2" long. About 3" from one end is a small "bump" on the side with a hole which is used to center this point on the center of the film after the film has been placed in the rectangular hole in the light box. Pivot the ruler on the center point until the edge intersects the center of a spot on the x-ray film. Read as the number on the ruler scale (short side), which is a scale from 1 to 5, with equally spaced markings. Let's say you read 3.2. Now take a pencil, and find 3.2 on the other, larger scale on the left side of the ruler. Make a mark on the drawing paper at this point. This is proportional to $\frac{h}{l}, y = \frac{k}{l}$ times a scale factor on the paper. Call this scale factor "p". You should mark off a grid spaced by p.

The ruler assumes that the crystal to film distance D was 4 cm *

3.2 on the film scale corresponds to a distance of S=3.2 cm from the film center to the spot. Since D=4 cm , and the x-ray is deflected through an angle 2θ:

$$\tan(2\theta) = \frac{S}{D} \approx \frac{3.2}{4} = 0.8$$

Knowing the tangent of twice θ, we can calculate the cotangent of θ. The easiest way to do this is via the trig relation

$$\cot\theta = \frac{1 + \sqrt{1 + (\tan 2\theta)^2}}{tan2\theta} \approx 2.85$$

Why do we want to find the cotangent of theta? Because it gives the projection of the normal to the scattering plane in the crystal, projected onto the film plane. In the example, the cotangent =2.85, and the projection onto the film would be D times this. On the ruler, there is a scale factor "p". We measure on the left scale that 3.2 corresponds to 15.7 cm, approximately. This means that p is about 5.5 cm - not very precise. Trying another point,we let S=5.0 cm. 5.0 on the right hand scale corresponds to 10.4 cm at on the left scale. Solving for p, we get 5.00 cm.

So we can safely consider that the ruler converts S, read in the units of the right hand scale to a distance $p\ \cot\theta$, read at the corresponding number on the left hand scale. p = 5 cm.

The direction cosines of the normal are

* This is marked on the bridge between the two halves of the ruler. Do not use the other ruler, marked D=5 cm. These rulers cannot be used with any other value of D except the one marked on the ruler.

$$\frac{h}{\sqrt{h^2+k^2+l^2}}, \frac{k}{\sqrt{h^2+k^2+l^2}}, \frac{l}{\sqrt{h^2+k^2+l^2}}$$

So the projection of this normal onto the paper, assuming the z axis is perpendicular to the paper is:

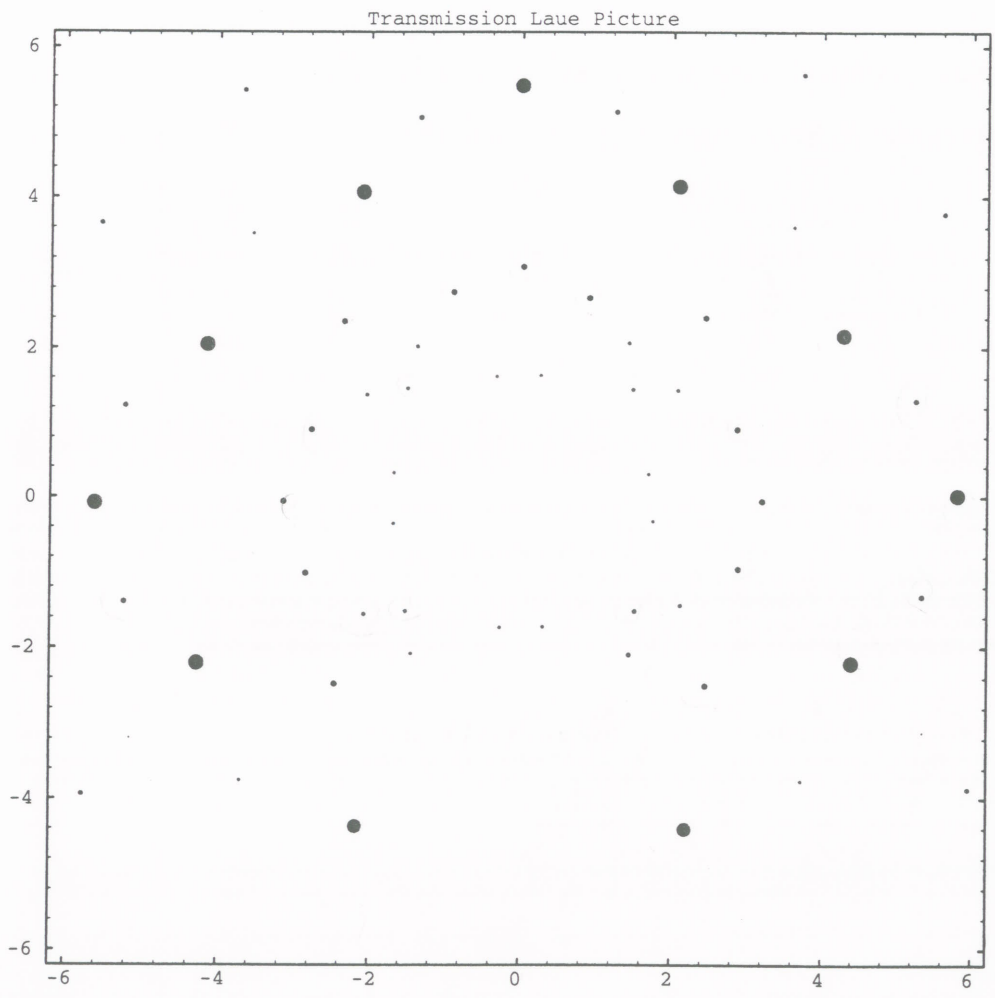
$$p\frac{h}{l}, p\frac{k}{l}$$

which are just the coordinates of the point you plot on your paper. In this way, you can directly plot the Miller indices. With careful plotting, you can identify most of your Laue spots this way with certain definite reflecting plane normals.

Analysis of X-3 Data

Part I: Transmission Laue Photographs

We assume that you have a well-aligned crystal for this analysis. Alignment is critical for the transmission photograph, and may not be good enough if you just read the scales on the goniometer— read the section on alignment! If the crystal is aligned, you should observe a high degree of symmetry in the x-ray picture. There should be symmetrical elliptical zones visible, as in the figure below. Try again if the picture shows misalignment. (In the actual picture, the most intense spots are elliptical. Intensities are only roughly estimated here. Some of the spots at the bottom were blocked by the crystal goniometer.)



Demystifying the Gnomonic Projection

A "gnomon" is "an object, such as the style of an sundial that projects a shadow used as an indicator". The word comes originally from the Greek root *gnomon*: one who knows, indicator, interpreter. This transformation is used in cartography. Unlike the stereographic projection used for the Wulff net (see Cullity, Chapter 2), the gnomonic transformation does not preserve angles. But it does turn ellipses into straight lines, and render the reciprocal lattice directly from the Laue diffraction pattern.

The Laue formula for diffraction is:

$$\vec{s} = \vec{s}_0 + \lambda \vec{B} \tag{1}$$

$\vec{s}$ is a unit vector along the direction of the scattered beam, $\vec{s}_0$ a unit vector along the incident beam direction, λ the x-ray wavelength, and $\vec{B} = (\frac{h}{a}, \frac{k}{a}, \frac{l}{a})$ is the reciprocal lattice vector, in this case for a cubic crystal of lattice constant a. h, k, l are three integers which define the normal to a plane of atoms in the lattice.

Squaring both sides, and solving for the wavelength, we obtain the formula:

$$\lambda = -\frac{2\vec{s}_0 \cdot \vec{B}}{\vec{B} \cdot \vec{B}} \tag{2}$$

Formulas (1,2) are equivalent to the Bragg formula:

$$\frac{2 \sin \theta_{hkl}}{\lambda} = \frac{1}{d_{hkl}} \tag{3}$$

θ is $\frac{1}{2}$ the x-ray scattering angle. d_{hkl} is the distance between planes with the same hkl normal. For a cubic crystal,

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \tag{4}$$

For a "centered" crystal, it can be shown that h+k must be an even number.

The Bragg formula, (3), is not as useful as (1,2) for the Laue experiment, because the scattering does not take place in a single plane, and there is not a single wavelength, but a range of wavelengths above a minimum wavelength determined by the voltage on the x-ray tube.

$$\lambda > \lambda_{min} = \frac{12.398}{V} \tag{5}$$

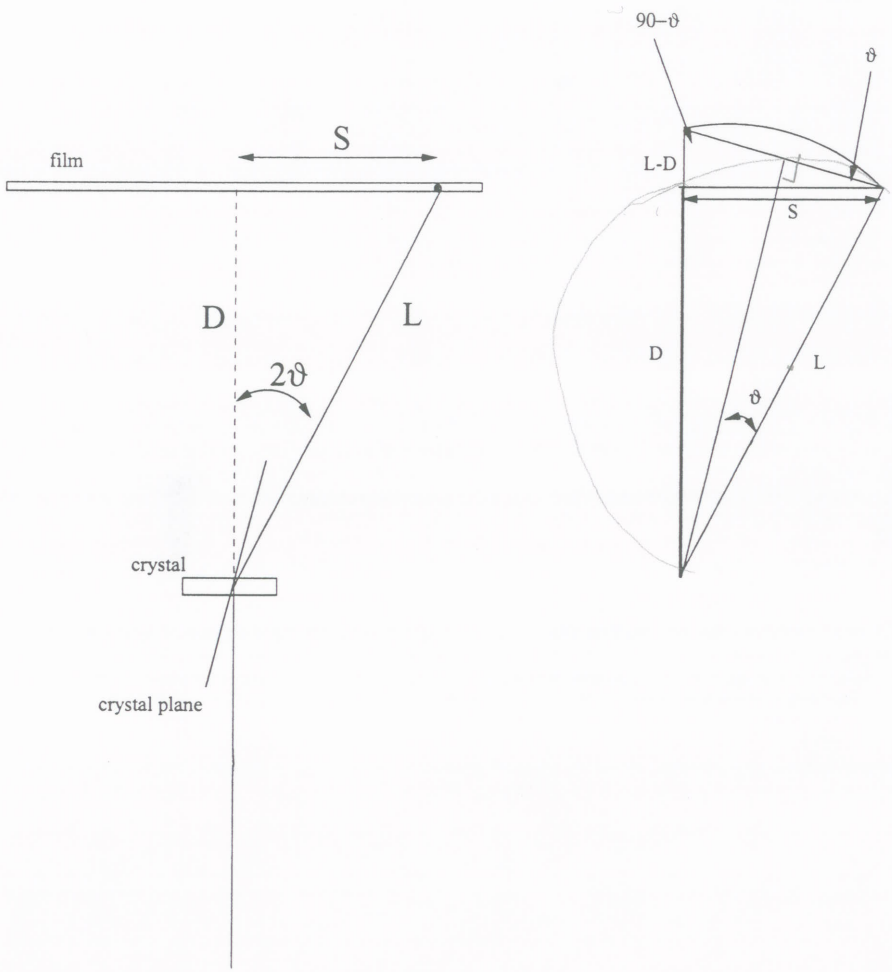
The units of wavelength in (5) are angstroms: Å (1 Å $= 10^{-8}$ cm). The units of voltage V are kilovolts (KV).

The figure below, on the left, shows the geometry of an x-ray reflection from a crystal plane, making a spot on the film. The x-ray beam is deviated by an angle 2θ. From the drawing, it is evident that

$$\tan 2\theta = \frac{D}{S}$$

The drawing on the right shows a geometrical construction which enables us to obtain $\cot\theta$. An arc of radius L is swung to the left until the radius is parallel to the section of length D– parallel to the undeviated x-ray beam. By comparing right triangles, the angle at the upper left is seen to be $90 - \theta$ degrees, so the angle on the right with the pointer label is θ. From this it is straightforward to see that

$$\cot\theta = \frac{S}{L - D}$$



All of the "magical" properties of the gnomonic projection follow from these geometric relationships. The student should check the formulas in the column on the right below. Of course, we have only proved

the usefulness of the gnomonic projection for cubic crystals with an x-ray beam perfectly aligned with one of the crystal axes.

Table I

hkl	$\bar{3}\bar{1}\bar{2}$	hkl
$h^2+k^2+l^2$	14	$h^2+k^2+l^2$
$\frac{\lambda}{a}$	$\frac{2}{7}$	$-\frac{2l}{h^2+k^2+l^2}$
$\vec{s}=\vec{s}_0+\lambda\vec{B}$	$(0,0,1)\ -\ (3,1,2)\frac{2}{7}=(-\frac{6}{7},-\frac{2}{7},\frac{3}{7})$	$\vec{s}=\frac{2l}{h^2+k^2+l^2}(-h,-k,h^2+k^2-l^2)$
distance crystal to film	4 cm	D
distance crystal to diffracted spot	$\frac{28}{3}$ cm	$L=\frac{h^2+k^2+l^2}{h^2+k^2-l^2}D$
x position of spot	$x_1=-\frac{6}{7}L=-8$ cm	$x_1=\frac{2hl}{l^2-(h^2+k^2)}D$
y position of spot	$y_1=-\frac{2}{7}L=-\frac{8}{3}$ cm	$y_1=\frac{2kl}{l^2-(h^2+k^2)}D$
$S=\sqrt{x_1^2+y_1^2}$=distance spot from center	$\sqrt{8^2+(\frac{8}{3})^2}=\frac{8}{3}\sqrt{10}$ cm	$S=\frac{2\lvert l\rvert\sqrt{h^2+k^2}}{\lvert h^2+k^2-l^2\rvert}D$
$\tan 2\theta=\frac{S}{D}$	$\frac{2}{3}\sqrt{10}$	$\frac{2\lvert l\rvert\sqrt{h^2+k^2}}{\lvert h^2+k^2-l^2\rvert}$
$\cot\theta=\frac{S}{L-D}$	$\frac{\sqrt{10}}{2}$	$Sign[h^2+k^2-l^2]\frac{\sqrt{h^2+k^2}}{\lvert l\rvert}$
$\cos(\pi+\phi)=-\frac{x_1}{S}$	$\frac{3}{\sqrt{10}}$	$\frac{h}{\sqrt{h^2+k^2}}$
$\sin(\pi+\phi)=-\frac{y_1}{S}$	$\frac{1}{\sqrt{10}}$	$\frac{k}{\sqrt{h^2+k^2}}$
$x_G\equiv p\cot\theta\cos(\pi+\phi)$	$\frac{3}{2}p$	$p\frac{h}{\lvert l\rvert}Sign[h^2+k^2-l^2]$
$y_G\equiv p\cot\theta\sin(\pi+\phi)$	$\frac{1}{2}p$	$p\frac{k}{\lvert l\rvert}Sign[h^2+k^2-l^2]$
$(\frac{x_G}{p},\frac{y_G}{p},1)$	$(\frac{3}{2},\frac{1}{2},1)$	$\pm(\frac{h}{\lvert l\rvert},\frac{k}{\lvert l\rvert},1)$

Reducing the fractions in the second column in the last row to whole numbers by multiplying through by 2, we establish that the hkl=312. (There is no difference between this and -3,-1, and -2, since they both define the same normal to a definite plane in the crystal.)

Notice that the gnomonic projection gives us the last three lines in Table I directly from the measurements on the paper construction, hence we can determine hkl for a particular spot. From this result, we deduce that

$$a=\frac{7}{2}\lambda$$

From what we know about tube voltage and the resultant cutoff on the x-ray spectrum, we can then deduce a lower limit on the lattice constant. If we examine more Laue diffraction spots, we can sharpen the limit on the lattice constant.

The gnomonic projection constructs the projected points x_G, y_G from the points x_1, y_1 by the rules

$$x_1 = D \tan 2\theta \cos \phi$$

$$y_1 = D \tan 2\theta \sin \phi$$

$$x_G = p \cot \theta \cos (\pi + \phi)$$

$$y_G = p \cot \theta \sin \pi + \phi)$$

The proof that we can deduce the reciprocal lattice point for an arbitrary hkl is shown in the table above, at least for an x-ray beam down the z axis.

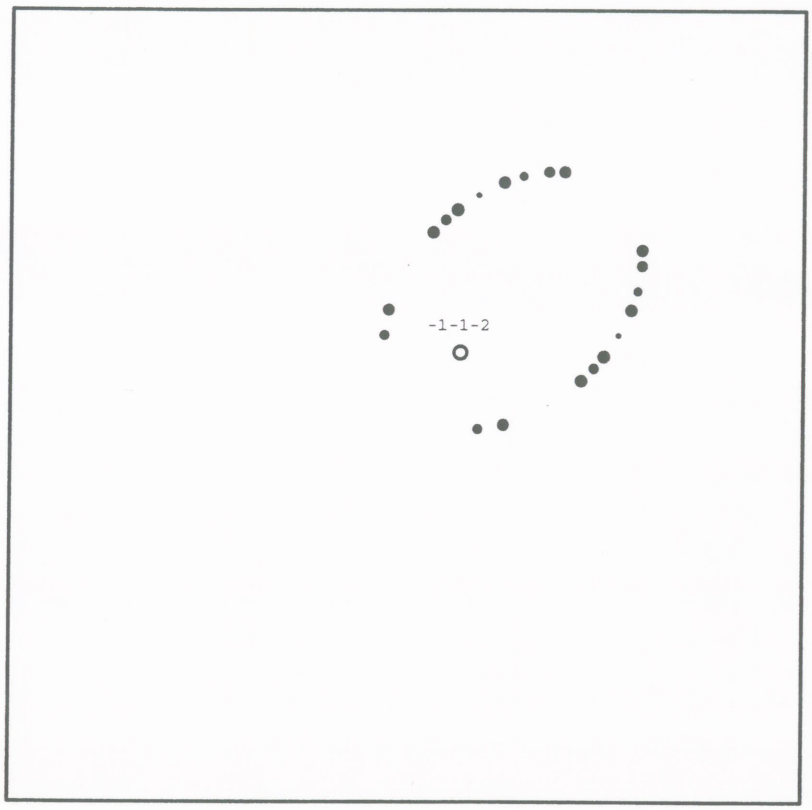
It would be a good idea to work through some of the numbers in the Table I, in order to get a better feeling for the theory.

Example Of Transmission Laue Spots

The figure below shows the simulation of an elliptical collection of spots of varying intensity on the film. The hollow circle, labelled "-1-1-2" does not actually represent anything visible on the film, but is the projection of the "zone", a direction which lies in all of the planes represented by the visible hkl, and is therefore normal to them. If you choose such a direction in the lattice, all of the atoms will lie on a line $ma(u, v, w)$, with m,u,v,w integers and a the lattice constant. (The direction is equivalent to 112.) Any plane containing the zone can diffract the x-ray beam, so the diffracted beams consist of circular cone of beams, one beam whenever hkl satisfies the condition of normality ($hu+kv+lw = 0$) and the Laue conditions for diffraction—in this case, whenever a wavelength exists to satisfy (1,2). If the film plane intersects this cone at an angle less than 45 degrees, an elliptical pattern of spots is generated, with the center of the film being on the ellipse. Think of a plane through a line (the zone). Rotate this plane around the line and record a spot whenever diffraction is possible.

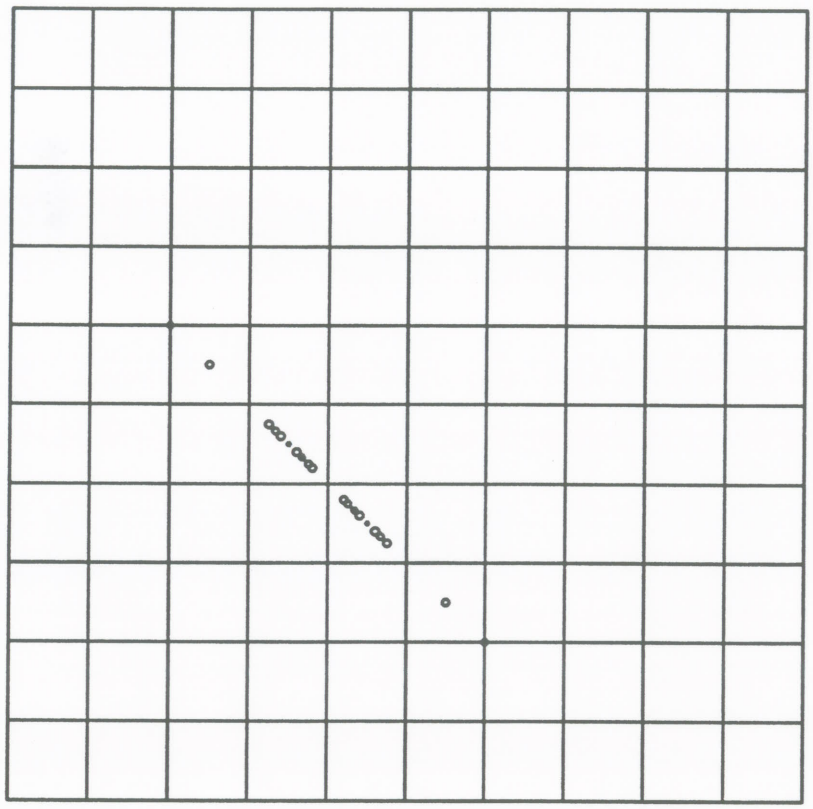
Of course the film actually produced in this experiment will have a more complicated pattern of spots, since there are many zones and therefore many ellipses (and straight lines and hyperbolae, depending on the angle of the zone direction from the film). Most zones will contribute only a few spots, however. Analysis should concentrate on the most intense spots.

fcc or bcc cubic crystal, lattice constant 3.3 angstroms
Mo spectrum, triangle approximation, 35. KV



The gnomonic projection of this set of spots appears below.

GNOMONIC PROJECTION
fcc or bcc cubic crystal, lattice constant 3.3 angstroms
Mo spectrum, triangle approximation, 35. KV



The spacing of the grid p is arbitrary. We see a point in the upper left side of the pattern at $x_G = -3, y_G = 1$. This must correspond to hkl= $\bar{3}11$, which is normal to 112, as it must be. In the lower right side of the graph a point appears at $x_G = 1, y_G = -3$. For this point, hkl = $1\bar{3}1$. The wavelength for diffraction is, assuming the crystal is cubic

$$\lambda = \frac{2a}{10} > \lambda_{min}$$

Therefore, $a > 5\lambda_{min} = 1.75$ $\mathring{A}$.

To see if we can get a better limit on the lattice constant, we look at some other points. The more intense points are apt to diffract with the most common wavelength in the spectrum, i.e. the shortest. Take the pair of points $x_G = -2.5, y_G = 0.5$ and $x_G = 0.5, y_G = -2.5$. This gives hkl = $\bar{5}12$ and $1\bar{5}2$ respectively.

In both cases

$$\lambda = \frac{4a}{30}$$

So

$$a > \frac{15}{2}\lambda_{min}$$

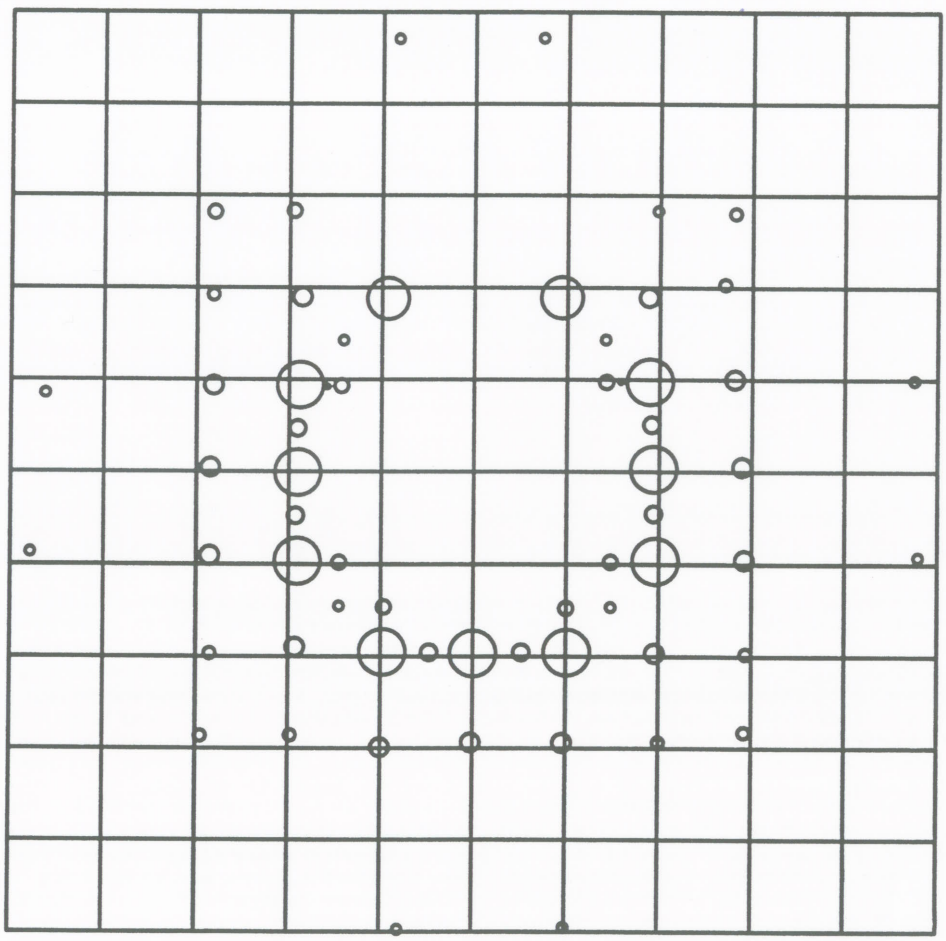
For 35 KV, using (5) above, $\lambda_{min} = 0.354\ \mathring{A}$. Hence the limit we obtain from these points is $a > 2.66\ \mathring{A}$.

Less intense points will not give a better limit, since fewer harmonics are present in the spectrum.

Gnomonic Projection Of Transmission Photograph

We can apply a gnomonic projection to the real data presented in the figure on page 1. From the picture, with a bit of allowance for residual effects of misalignment, we can classify the lines by hkl and intensity †. Some lines were shadowed by the crystal mount or goniometer.

GNOMONIC PROJECTION OF DATA ON PAGE 1



† Based on subjective guesses.

Table II: Results of Gnomonic Projection

Intensity	Miller Indices	Comments
Very Strong	$121, 211, 201, 2\bar{1}1, 1\bar{2}1, 0\bar{2}1, \bar{1}\bar{2}1, \bar{2}\bar{1}1, \bar{2}01, \bar{2}11, \bar{1}21$	021 in shadow
Strong	$221, 412, 4\bar{1}2, 2\bar{2}1, 1\bar{4}2, \bar{1}\bar{2}1, \bar{2}\bar{2}1, \bar{4}\bar{1}2, \bar{4}12, \bar{2}\bar{2}1,$	
	$\bar{3}11, \bar{3}01, \bar{3}\bar{1}1, 3\bar{1}1, 301, 311, \bar{1}\bar{3}1, 0\bar{3}1, 131$	
Medium	$231, \bar{2}31, 3\bar{2}1, 2\bar{3}1, \bar{3}\bar{2}1, \bar{3}21, \bar{3}31, 3\bar{3}1, \bar{3}, \bar{3}\bar{3}1$	
Weak	$\bar{5}11, \bar{5}, \bar{1}1, 511, 5\bar{1}, 1, \bar{1}51, 151, 1, \bar{5}1, \bar{1}\bar{5}1$	
	$332, \bar{3}32, \bar{3}\bar{3}2, 3\bar{3}2$	
	$322, 2\bar{3}2, 3\bar{2}2, \bar{2}\bar{3}\bar{2}, \bar{3}\bar{2}2, \bar{3}22$	

So we have the sets $\{201\}, \{121\}$ for the strongest reflections and $\{122\}, \{013\}, \{113\}, \{124\}$ for the next strongest. "Medium" includes $\{223\}$,$\{133\}$,$\{123\}$. Weak reflections include $\{115\}$ and $\{233\}$. The very lowest orders: $\{001\}, \{111\}$ are not visible on the film. They would be at large distances from the center.

The perfect four-fold symmetry suggests that the crystal is cubic, with a $\frac{c}{a}$ ratio of one. Assuming that is true, we can use (2) to solve for the ratio of x-ray wavelength to lattice constant a:

$$\frac{\lambda}{a} = \frac{2l}{h^2 + k^2 + l^2}$$

From the list of observed (and absent) reflections above in Table II, we can construct Table III below. Remember that all harmonics of the x-ray which are allowed, down to the shortest wavelength present here, 0.354 Å. The more harmonics, the stronger the reflection.

Table III: Ratios Of Wavelength To Lattice Constant

Generic Miller Index	Observed Example	$\frac{\lambda}{a}$	Comment
012	201	$2/5 = 0.4$	very strong
112	211	$1/3 = 0.33$	"
122	221	$2/9 = 0.22$	strong
013	301	$1/5 = 0.2$	"
113	311	$2/11 = 0.19$	"
124	412	$4/21 = 0.19$	"
123	321	$1/7 = 0.14$	medium
223	322	$4/17 = 0.24$	"
133	$3\bar{3}1$	$2/19 = 0.11$	"
115	$1\bar{5}1$	$2/27 = 0.07$	weak
233	332	$2/11 = 0.18$	"
114	(411)	$1/9 = .11$	not seen
124	(421)	$2/21 = .095$	"
125	(521)	$1/15 = .067$	"
134	(431)	$1/13 = .077$	"

In the table above, the reflections are grouped according to decreasing order of (subjective) intensity. A very strong line implies that many harmonics are possible, while a weak line implies that the lowest harmonic is already close to the minimum wavelength.

With only two exceptions, 233 and 223, our subjective assignment of intensities follows the decreasing order of $\frac{\lambda}{a}$ exactly. If the lattice constant had been smaller than $\frac{.354}{.07} = 4.8\ \mathring{A}$, the $\{1\bar{5}1\}$ lines and three others like it would not have been seen. We need to have $a < \frac{\lambda_{max}}{0.4}$, so $\lambda_{max} > 1.9\ \mathring{A}$, which is reasonable.

Other lines do not shed much light on the maximum value of the lattice constant. We can get a rough estimate from the rapid decrease in intensity going from the "very strong" group to the "strong" group. This means that, for example, $0.2a < .354\ \mathring{A}$, so $a < 1.8\ \mathring{A}$. This contradicts our limit of 4.8 Å, so a selection rule for the structure factor could be instead the reason for the decrease in intensity.

The lines not seen also could be due to the structure factor, so it is hard to get an upper limit on the lattice constant. If we assume the 521 line was excluded by the minimum wavelength cutoff, we get an estimate $a_{max} < \frac{.354}{.067} = 5.3\ \mathring{A}$.

More about the crystal could perhaps be learned from the relative intensities, including the absence of some lines with otherwise observable $\frac{\lambda}{a}$ ratios, like 411.

One word of warning: all of Table III was based on the assumption that the crystal is a cubic lattice.

All bets are off if it is not, since the ratio $\frac{\lambda}{a}$ will depend on the $\frac{c}{a}$ ratio. We assume that four-fold symmetry means the lattice is cubic.

Part II: Back Reflection Photograph

Analysis Of Back Reflection X-ray Data

In this experiment, you observe the back reflected x-rays from a silicon crystal placed 3 cm from the film. The goal is to identify the hkl values for the most intense spots on the film, and find out the crystal orientation. This method is commonly used in the Snee Hall X-ray Diffraction Facility to orient crystals precisely.

There are two references, at least one of which must be read before this part of the experiment can be understood and analyzed. The best reference is the book by B.D. Cullity, "*Elements of X-ray Diffraction*". Another discussion of back reflection Laue photographs appears in B.E. Warren's book *X-ray Diffraction*, pages 77-83. We will keep the discussion brief here †.

Chapter 8 in Cullity's book has the key information. This part is reprinted in the Physics 410-510 X-3 writeup. (The first edition was in 1956 and the second in 1978, but little change occurred in the part of interest here.) The key figures to study and understand are figures 8-1 and 8-2. In figure 8-1, imagine a family of crystal planes all of which are orthogonal to a line of atoms in the crystal. The line of atoms is called a "zone axis" (abbr. "Z.A."). Zone axes are denoted by square brackets. For example, the line of atoms along the z axis is [001], along the x axis is [100], etc. In general, each such zone axis is uniquely described by a set of integers u,v,w: $[uvw]$. Crystal planes described by Miller indices (hkl) which are orthogonal must obey the relation $hu + kv + lw = 0$. In figure 8-1, the lines with arrows are the reflections by these planes. The reflections describe a space cone, and the projection on the film is a hyperbola of spots, each of which satifies the Laue conditions for reflection. So what you observe on the film are collections ("zones") of spots which form one branch of a hyperbola. Usually, between 6-10 such hyperbolae can be traced out from the film.

Consider the diagram below (taken from Warren's book, page 79):

† These books are available in the P410/510 Library.

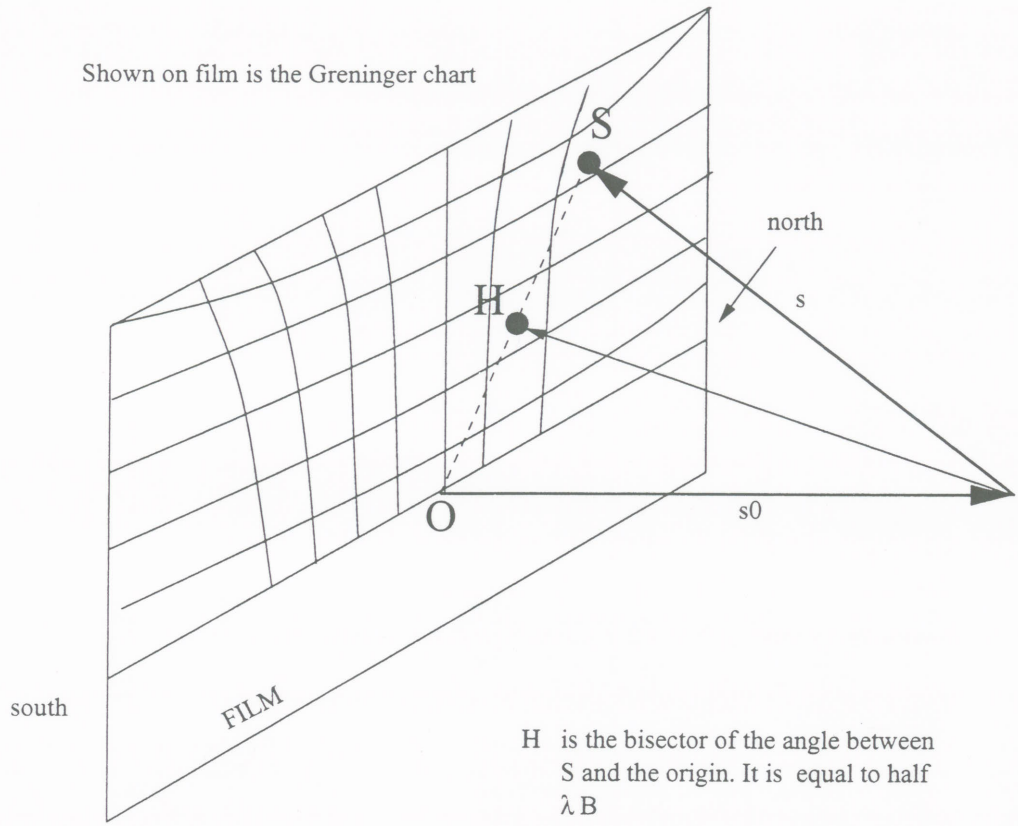


Fig 6.7, page 79 in book by B.E.Warren

The Greninger chart simply gives a way to get the spherical polar coordinates of $\lambda\vec{B}$ (normal to the reflecting plane from the xy position of the back-reflected spot S on the film. The angle $\gamma(x, y)$ is "longitude" or azimuth and the angle $\delta(x, y)$ is the "latitude" or 90° minus the polar angle in spherical polar coordinates. The geometry of these angles is illustrated in Cullity's figure 8-2. By rotating the film against the Greninger chart (see page 78, Warren), you can get the spatial angle between the reciprocal lattice vectors of any two spots on the film. Warren advises using the three most intense spots (these will be the lowest hkl vectors), obtaining the angles between them, and then making the identification using the table for crystal with the diamond structure, which we have reproduced below. This method may not work well, however, so it is probably more useful to proceed more methodically (unless you are lucky with the 3 spot method). γ, δ are measured as accurately as possible, within 0.5°. These values, for each reflection point, are to be plotted on a stereographic projection (Wulff chart). Xerox copies of Wulff charts are available for this. Accuracy is essential here. In the general case, all points on a specific zone will give a great circle on the Wulff chart. (Read up on stereographic projections in Chapter 2 of Cullity.) Then, rotate the transparent Wulff chart until the points line up on a line of constant γ. If they can't be made to do this by a simple rotation of the Wulff chart, they do not belong on a great circle, hence not to the same zone. Trace all the great circles and

mark out the point which would be the "North Pole" if the great circle were rotated to lie on the equator (on a 3D sphere). You can use the rotated Wulff chart, along the line $\delta = 0$ to find the unique point 90° away from zone points, i.e. with a latitude $\gamma_{Z.A.} = \gamma + 90°$. This is the zone axis. Finally, measure the angles between a point common to two zones and the zone axes to find a 90° spherical triangle (in 3D on the sphere, this is the projection of it.) In one measurement, we found two such triangles.

Now refer to the standard (001) projection of a cubic crystal (figure 2-37 in Cullity or available in transparent form in the lab. From this you can identify candidates for the 001 point and hence determine the misalignment of the crystal. After doing this, you can determine the Miller indices of most of the spots in your picture, by looking at the angles.

All this can be done manually, as was intended before computers were available, or in the computer. Either way leads to satisfactory results. See the Appendix I for the formulas for the various transformations you will need if you write a program.

Selection Rules on hkl for silicon

Silicon has the diamond structure—two interpenetrating face-centered cubic lattices. This is discussed in "*Solid State Physics*", by N. Ashcroft and N.D. Mermin, page 79. The selection rules † are: ($n_1 n_2 n_3 = hkl$):

Crystal Type	Selection Rule
face-centered cubic	h,k,l either all even or all odd
diamond	" plus either h+k+l=odd or else 4 n,n=integer

The table of angles for allowed hkl requires an explanation. (001) is forbidden for an fcc lattice, (002) is allowed for fcc (all even), but forbidden for the diamond structure, which is silicon. However, (004) is allowed, provided that the necessary wavelength is present in the continous spectrum of the Laue experiment. The program which calculated the angles in the page below considers the selection rules, but divides out the greatest common multiple of the hkl. So (004) becomes (001) and is listed as such.

Also, if we mean the family of all Miller indices equivalent by symmetry to, say (111), we write {111}. This would be the set of planes with Miller indices $(111), (\bar{1}11), (1\bar{1}1), (11\bar{1})$. $(\bar{1}\bar{1}\bar{1})$ is exactly the same direction as (111),so we dont consider this as separate.)

This explains why the table below says that the (111) normals can make angles of $0°, 70.5°$ with each other. The unit vectors of two members of this group are $\frac{(1,1,1)}{\sqrt{3}}, \frac{(\bar{1},1,1)}{\sqrt{3}}$. The cosine of the space angle between them is given by the scalar product of the two unit vectors:

$$\cos\Phi = \frac{1}{3}$$

† See Ashcroft and Mermin, page 106.

From a table of arc-cosines, we can see that $\Phi = 1.23096$ radians i.e. $\Phi = 70.529°$.

The reduction of the order by division out of the greatest common divisor in the hkl makes looking up things rather difficult in the table. We found it easiest to ignore the common factor and treat (400) as if it were (100), keeping in mind that only the (400) (and higher multiples of it) can actually occur in the photograph.

Possible Angles Between Planar Normals For Diamond Lattice

"533____" indicates the angles between all members of the {533} group and all members of the groups listed with colons below it. For example, the possible angles between a 533-type plane and a 111-type plane are $14.4°, 63.9°, 84.9°$. Angles greater than 90° are not listed.

111____
111 : 0. 70.5
110 : 35.3 90.
100 : 54.7

110____
110 : 0. 60. 90.
100 : 45. 90.

311____
111 : 29.5 58.5 80.
110 : 31.5 64.8 90.
311 : 0. 35.1 50.5 63. 84.8
100 : 25.2 72.5
211 : 10. 42.4 60.5 75.7 90.

331____
111 : 22. 48.5 82.4
110 : 13.3 49.5 71.1 90.
311 : 25.9 40.5 51.5 61. 69.8 78.
331 : 0. 26.5 37.9 61.7 80.9 87.
100 : 46.5 76.7
211 : 20.5 41.5 68. 79.2

100____
100 : 0. 90.

211____
111 : 19.5 61.9 90.
110 : 30. 54.7 73.2 90.
100 : 35.3 65.9
211 : 0. 33.6 48.2 60. 70.5 80.4

Possible Angles (continued)

511

111 :	38.9	56.3	70.5				
110 :	35.3	57.	74.2	90.			
311 :	9.45	29.5	41.	58.5	66.	80.	86.7
331 :	33.	41.4	55.	60.9	77.2	87.5	
100 :	15.8	78.9					
211 :	19.5	38.2	51.1	61.9	71.7	81.	
511 :	0.	22.2	31.6	66.	70.5	87.9	

531

111 :	28.6	46.9	73.	84.4									
110 :	17.	44.2	61.4	76.2									
311 :	14.5	30.	40.1	48.5	55.9	75.2	81.2	87.1					
331 :	14.2	26.9	35.5	48.8	54.4	64.8	69.6	74.2	78.8	87.8			
100 :	32.3	59.5	80.3	211 :	15.	34.1	46.4	56.5	65.5	74.	82.1	90.	
511 :	19.4	28.6	41.6	46.9	51.8	65.	69.	73.	76.8	84.4			
531 :	0.	19.5	27.7	34.	44.4	48.9	57.1	60.9	64.6	68.2	75.1	78.5	88.4

533

111 :	14.4	63.9	84.9							
110 :	30.4	49.7	77.5	90.						
311 :	15.1	38.6	46.4	59.6	65.6	71.2	87.4			
331 :	19.2	36.4	42.7	62.9	71.6	79.9	84.			
100 :	40.3	62.8								
211 :	5.05	29.4	51.5	60.1	75.6	82.8				
511 :	24.5	42.8	47.5	56.1	60.1	67.6	78.1			
531 :	17.5	31.7	37.	41.6	45.9	60.7	70.4	73.5	85.6	88.5
533 :	0.	24.9	54.5	60.8	77.9	80.6				

Summary Of Steps In Back Reflection Analysis

1. Trace film spots on tracing paper. Sketch possible hyperbolae on this paper and number the points. Follow the instructions in Cullity for maintaining the orientation of the film using the cut corner.

2. Using the Greninger chart, find γ, δ for each point. Both angles can range from $-\frac{\pi}{2}$ up to $\frac{\pi}{2}$. If you are using the computer, digitize the x,y points, transform to Cullity's xy coordinates, and compute γ, δ for the planar normals from the formulas in the appendix.

3. Plot these points on the Wulff chart. Label with the numbers and trace possible great circles, which you check by superimposing a rotated transparent Wulff chart. (This can be done in the computer too, if you wish, but it would take too long to describe the procedure here. Ask your instructor.)

4. Find the corresponding zone axes. It is traditional to mark these with open circles, and label them with letters: "A","B", etc.

5. Find at least one spherical right triangle (three 90° angles) which contains two zone axes and one pole (planar normal).

6. Try out the hypothesis that the pole is (001). (We are saying that we do know the silicon crystal is aligned not too far from that axis.) If that is not confirmed by consistency of angles between other points and the 001 point, find another spherical right triangle. If necessary, add more points.

7. After confirming which point is the 001 point, try to identify hkl for as many points as you can, using the standard projection(001) chart as your guide.

Appendix I

Mathematical Formulas For Transformations

Greninger Chart (X,Y,D in centimeters)

$$D = 3$$

$$S = \sqrt{X^2 + Y^2}$$

$$L = \sqrt{S^2 + D^2}$$

$$\theta = \tan^{-1}\left[\frac{L - D}{S}\right]$$

$$\tan\gamma = \left(\frac{Y}{S}\right)\left(\frac{L - D}{S}\right)$$

$$\sin\delta = -\left(\frac{X}{S}\right)\sin\theta$$

$$\gamma = \tan^{-1}(\tan\gamma)$$

$$\delta = \sin^{-1}(\sin\delta)$$

Spherical Polar Coordinates For Unit Sphere

To agree with Cullity, the coordinate system is unusual: $+x$ is horizontal, to the right, $+y$ is vertical on paper, $+z$ is out of paper.

$$\theta_{polar} = \frac{\pi}{2} - \delta$$

$$x = \cos\theta_{polar}$$

$$y = \sin\gamma \ \sin\theta_{polar}$$

$$z = \cos\gamma \ \sin\theta_{polar}$$

Stereographic Projection Onto Wulff Chart From γ, δ

X_W refers to the horizontal direction on the plane of the Wulff chart, Y_W to the vertical direction. Varying γ, δ over the allowed region from $-\frac{\pi}{2}$ to $\frac{\pi}{2}$ gives a stereographic projection of the hemisphere corresponding to positive x in spherical polar coordinates. The sphere has unit radius.

$$X_W = \frac{\sin\delta}{\sqrt{1 + \cos\delta\cos\gamma}}$$

$$Y_W = \frac{\cos\delta\sin\gamma}{\sqrt{1 + \cos\delta\cos\gamma}}$$

Notice that the formulas are not symmetric in γ, δ. This is correct.