The Use of X-Rays for Determining the Orientation of Quartz Crystals

By W. L. BOND And E. J. ARMSTRONG

THIS paper is one of a series by the Crystal Research Group on the manufacture of quartz oscillator plates. Certain sections of it which are not original, but rather adaptations of text book material to the present problem, are included for purposes of completeness and for the convenience of those readers whose knowledge of the crystallographic literature may be limited.

3.1 PRODUCTION OF X-RAYS FOR QUARTZ CRYSTAL X-RAY WORK

X-rays are produced when electrons strike a metal target at high velocity. The wave-length of X-rays given off from an X-ray tube varies from the longest which can pass through the X-ray tube window to the shortest that can be produced from the given target by the applied peak voltage. By analogy to the visible spectrum this is referred to as "white" radiation. For each different metal, however, there are characteristic radiations of certain wave-lengths whose intensity markedly exceeds those of other wave-lengths (Fig. 3.1). The strongest of these characteristic radiations is known as the $K\alpha_1$, the next strongest (generally half as strong and of slightly longer wave-length) as $K\alpha_2$ and the third strongest (shorter in wave-length than $K\alpha_1$) is $K\beta$. The higher the atomic number of the target, the shorter will be the wave-length of the characteristic radiation. Therefore higher voltages will be required to excite the characteristic radiation from the heavier metals. (The minimum wave-length of X-rays that can be excited by any given voltage is given by the equation $\lambda_{min.} = \dfrac{1.234 \times 10^{-4}}{V}$ where V is expressed in volts and $\lambda_{min.}$ in Ångstrom units).

Higher voltages also raise the intensity of the white radiation and, at any given voltage, the white radiation produced from a heavy metal target is more intense than that produced from a lighter metal target (see Figure 3.1). When "white" radiation is desired, as in Laue photography, heavy metal targets, such as tungsten, are used: when "monochromatic" radiation is desired, as in crystal goniometry, the lighter metal targets, such as copper, are used because, with a lighter metal target (wave-length of characteristic radiation long) the voltage, and therefore the intensity of the

white radiation, cannot be raised very high before exciting the characteristic radiation whereas, with a heavy metal target (wave-length of characteristic radiation short) considerable intensity of white radiation can be produced without exciting the characteristic radiation. This is illustrated in Fig. 3.1 which shows that a potential of 35,000 volts is high enough to excite the K group radiation from molybdenum, but not high enough to excite the shorter wave-length K radiation from tungsten which, further, gives more intense white radiation at this voltage. Even higher voltages, resulting in
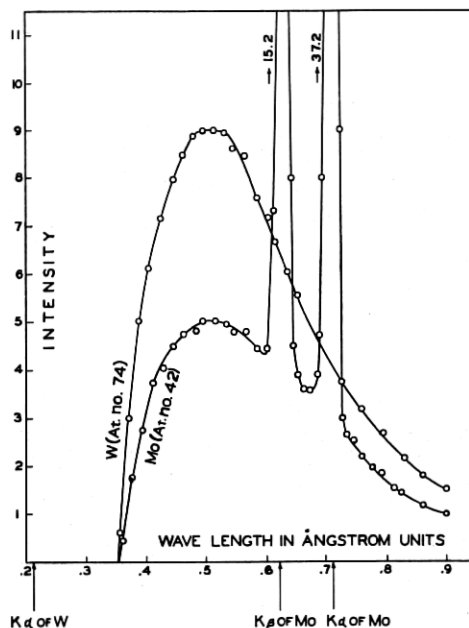

Fig. 3.1—Variation of intensity with wave-length of X-rays from tungsten and molybdenum targets at 35,000 volts

more intense white radiation, could be used with tungsten without exciting the characteristic radiation (λ = .209).

Figure 3.2 shows the I-λ curve (estimated) for copper, the target metal commonly used in quartz X-ray work, which has a small atomic number and can therefore be used as a source of "monochromatic" X-rays with moderate voltages. (A further advantage of copper for quartz work is pointed out at the end of this section).

The $K\alpha_1$ and $K\alpha_2$ wave-lengths are so close together that for most uses of "monochromatic" radiation no attempt is made to eliminate the $K\alpha_2$ radiation. The $K\beta$ radiation, however, gives a distinct intensity peak of shorter wave-length which must be reduced as much as possible by use of a metal filter having a high absorption coefficient for the $K\beta$ radiation of

the target used. In most cases the best filter-metal for the $K\beta$ radiation is the second element below the target metal in the periodic table. For example, a nickel filter 0.0005 inches thick is used with the copper target which is used for X-ray goniometry of quartz.

The minimum voltage that will excite the $K\alpha_1$ radiation of copper (wavelength $\lambda = 1.5374$A) is 8.86 kv., but a voltage of 30 or 40 kv. is usually used to obtain adequate intensity.
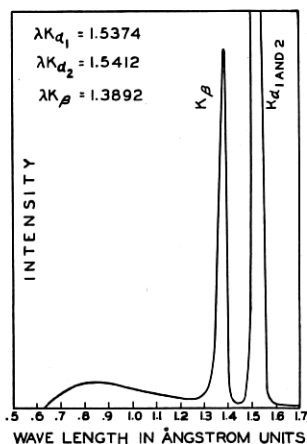

Fig. 3.2—Variation of intensity with wave-length of X-rays from a copper target at 20,000 volts (estimated)

3.2 DETECTION OF X-RAYS

X-rays may be detected by means of heat effects, fluorescence of appropriate screens, photographic effects and by ionization of gases.

Heat measuring devices are not suited to routine intensity measurements. The other three means of X-ray detection are all being used for various types of quartz work but the ionization of gases is used most widely.

The ionization method involves a chamber which consists essentially of a gas-filled metallic cylinder containing an electrode. A potential is maintained between the electrode and the cylinder so that when the gas is ionized by the X-rays the positive and negative ions produced are drawn to the oppositely charged electrodes, thus constituting an electric current, which current is proportional to the intensity of the X-rays entering the chamber. This current is commonly indicated by a current meter preceded by a special vacuum tube amplifier.

The gas used in the chamber is often air but methyl bromide is about 30 times more readily ionized, xenon 155 times. The only advantage of air is that the chamber need not be gas-tight. The entrance to a sealed ionization chamber must be closed by a substance which does not result in undesirable absorption of the X-rays. Since a nickel filter is needed

somewhere in the path of the X-rays to absorb the $K\beta$ radiation from the copper target, it may be used here to seal the entrance of the chamber.

The Geiger-Müller counter is similar to the ionization chamber, but is operated at such high voltage that the gas is always near breakdown, the passing of X-rays supplying the impetus to complete the breakdown. Because of the high voltage the ionization continues to be strong as the intensity of the radiation declines. Such a lag is likely to cause erroneous maximum readings in crystal measurement.

3.3 PHYSIOLOGICAL EFFECTS OF X-RAYS

Even a few minutes direct exposure to X-rays from such a source as the G. E. CA6 tube will result in a burn that will become apparent in a day or so. In most cases such a burn, if not repeated, will heal without ill effects, but because the physiological effect of X-rays is cumulative, repeated exposure to direct radiation could result in a burn that would not heal and might become cancerous.

Pinhole leaks which permit direct radiation to escape are thus exceedingly dangerous. They may be detected by use of a fluorescent screen in a darkened room. A sheet of lead-glass $\frac{1}{4}$ to $\frac{1}{2}$ inch thick should be held between the fluorescent screen and the observer.

X-rays are scattered from all substances which they strike and repeated exposure to this scattered radiation may result in a harmful decrease in the white blood corpuscles, in sterility, and perhaps in serious burns.

To test for scattered radiation dental X-ray films should be arranged as close to the instrument on all sides as any part of the operator's body can get and left for a period of two weeks of normal operation. If there is no position in which the film becomes fogged during the two weeks, the operator is safe. A narrow lead strip across the film will provide an un-exposed portion for comparison with the exposed portion. It should be emphasized that a dental film carried in the pocket is an inadequate safety test, since the hands are frequently the most dangerously exposed part of the body.

The absorptive power of shielding materials is proportional to the density of the material. Minimum adequate shielding is provided by 1.5 mm. or about $\frac{1}{16}$ inch of lead or its equivalent for protection against X-rays generated at 70 kv.[1] Equivalent thicknesses of shielding materials are as follows:

Lead	$\frac{1}{16}$ inch
Lead rubber	$\frac{1}{8}$–$\frac{1}{4}$ inch
Lead glass	$\frac{3}{8}$–$\frac{1}{2}$ inch
Steel	$\frac{1}{2}$ inch
Bricks and concrete	6 inches
Woods	60 inches

[1] Davey, W. P., "Study of Crystal Structure and its Applications," McGraw-Hill, 1934.

The United States Bureau of Standards has issued a 28-page booklet entitled "X-Ray Protection" which may be obtained from the Superintendent of Documents, Washington, D. C. for 10 cents.

3.4 Diffraction of X-Rays by Crystals

Following the work of the French crystallographer Haüy at the end of the eighteenth century the theory that crystals were made up of small identical units in orderly arrangement was widely held by crystallographers. In the late nineteenth century these units were thought of as intersecting planes of atoms. Then, in 1912, the German physicist von Laue conceived the idea that such a lattice of atoms should act as a three-dimensional diffraction grating for electromagnetic waves of wave-length approximating the interplanar spacing of the lattice. If X-rays were, as was suspected, electromagnetic vibrations of short wave-length, they might be of the right order of magnitude to obtain diffraction from crystals. When the experiment was tried it was found that a beam of X-rays (not monochromatic) passing through a crystal produced an orderly arrangement of spots on a photographic film, the type of photograph now known as a Laue photograph.

The term *reflection* may be used in place of *diffraction* since X-ray diffraction is like light reflection in that the entering and leaving beams make equal angles with the reflecting or diffracting atomic planes.[2] Since this concept is simpler, X-ray diffraction is commonly referred to as reflection.

Unlike light reflection, X-ray reflection can take place only under the conditions given by the following equation which is known as the Bragg law

$$n\lambda = 2d \sin \theta \tag{3.1}$$

where n = a small whole number,
   λ = wave-length of X-rays used (generally stated in Ångstrom units)
   d = distance between the atomic planes (generally stated in Ångstrom units).
   θ = angle between the X-rays and the atomic planes ("The Bragg Angle").

That is, the angle of incidence must be such that the path-length of two rays reflected from different atomic planes differs by a whole number of wave-lengths so that the emerging rays will be in phase. If the difference in path-length of the two rays is one wave-length the reflection is called the first-order reflection ($n = 1$). At some larger θ angle the path difference will be exactly 2λ and reflection will occur again. This is the second order reflection ($n = 2$). Monochromatic X-rays are used so that only

[2] Compton, A. H. and Allison, S. K., "X-Rays in Theory and Experiment," D. Van Nostrand, New York, 1935, Pages 340–346,

one value of θ will satisfy the equation for each order reflection. Thus, with a monochromatic X-ray beam one may not obtain reflection from the planes at all angles as with visible light, but only at such specific angles as satisfy the Bragg law. Further, even with optimum conditions for reflection the ratio of the intensity of the reflected X-ray beam to that of the incident beam is of the order of 1:10,000. The Bragg angle θ is highly critical and the reflection of X-rays from atomic planes therefore serves as a precise method of crystal orientation.

Figure 3.3 is a diagrammatic representation of the relation $n\lambda = 2d \sin \theta$. On such a diagram the following laws of X-ray reflection become obvious:

(1) λ must be smaller than $2d$, that is, the wave-length of X-rays used must be less than twice the inter-planar spacing of the atomic planes to be X-rayed.

(2) The number of different orders of reflection n obtainable from atomic planes with interplanar spacing d is fixed by the expression $n\lambda < 2d$. In
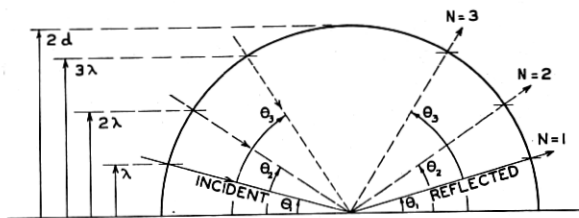

Fig. 3.3—A diagrammatic representation of Bragg's law, $n\lambda = 2d \sin \theta$

other words, since $\sin \theta$ cannot be greater than 1 the value of λ must be less than $\dfrac{2d}{n}$. The distance between the atomic planes parallel to the hexagonal prism of quartz is 4.2466 Å[3] and the wave-length of the $K\alpha_1$ radiation from a copper target is 1.5374 Å. Hence no reflection higher than the 5th order could be obtained from this set of planes using a copper target.

On the other hand, a target metal whose characteristic wave-length is very much shorter than $2d$ is undesirable since it gives so many orders of reflection from each set of atomic planes that the multitude of closely spaced reflections leads to confusion.

(3) Higher orders of reflection occur at larger θ angles.

(4) The relation of θ to λ is not linear but sinusoidal.

The reflected beam can only lie in a plane containing the normal to the atomic plane and the incident beam. Conditions are unchanged by rotating the crystal about the normal to the atomic plane being used.

[3] R. B. Sosman, "The Properties of Silica," Chemical Catalogue Co.. New York 1927.

3.5 THE NAMING OF ATOMIC PLANES IN CRYSTALS

It is convenient to be able to refer to any atomic plane in a crystal by some symbol that uniquely defines its orientation. The symbols commonly used for this purpose are known as Miller indices (or Bravais-Miller indices for the hexagonal system) and are the reciprocals of the intercepts of the atomic plane on a set of crystallographic axes chosen in accordance with the symmetry of the crystal. In quartz this set of axes is as shown in Fig. 3.4: a vertical axis c and three horizontal axes at 120° a_1, a_2, a_3.

Measurement of the interfacial angles of thousands of quartz crystals has shown that the natural faces have intercepts on the crystallographic
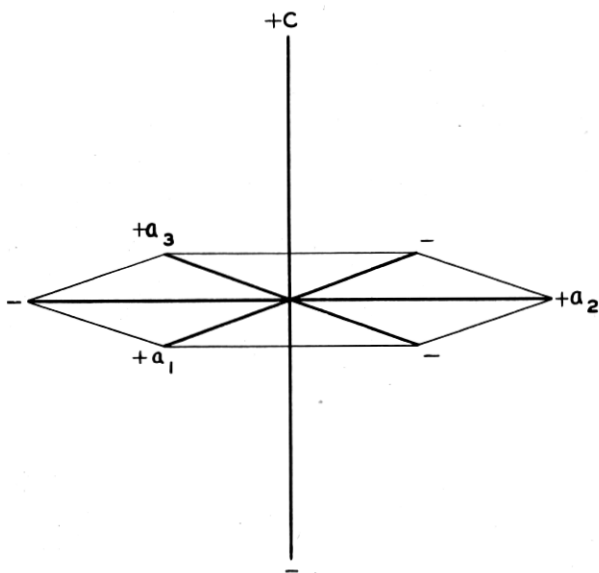

Fig. 3.4—Hexagonal crystallographic axes

axes that are integral multiples of a fixed distance, which is the same in the case of all three a axes and different in the case of the c axis. This fixed distance along the c axis is found to be 1.09997 times the fixed distance along the a axes. Therefore, the "unit length" of each of the a axes is said to be 1; that of the c axis 1.09997 and a face that cuts the c axis at 1.09997a from the origin is said to have the c intercept of 1. (This unit axial length is different for different substances but the same for all crystals of the same substance.) For example, the front cap face in figure 3.5 has the axial intercepts 1, ∞, −1, 1, naming the axes in the order a_1, a_2, a_3, c. The indices for this face are written (10$\bar{1}$1) (general form $hki l$). The front vertical face has the intercepts 1, ∞, −1, ∞ and the

indices $(10\bar{1}0)$. The first two digits of the indices of any vertical prism plane are the same as those of the adjacent cap faces: the final digit is always zero because they are parallel to the Z (or c) axis. Since the intercepts on a_1 and a_2 uniquely determine the intercept on a_3, the third digit of the symbol may be omitted. The omission is sometimes indicated by a dot, as $(10\cdot1)$ (general form $hk\cdot\ell$).
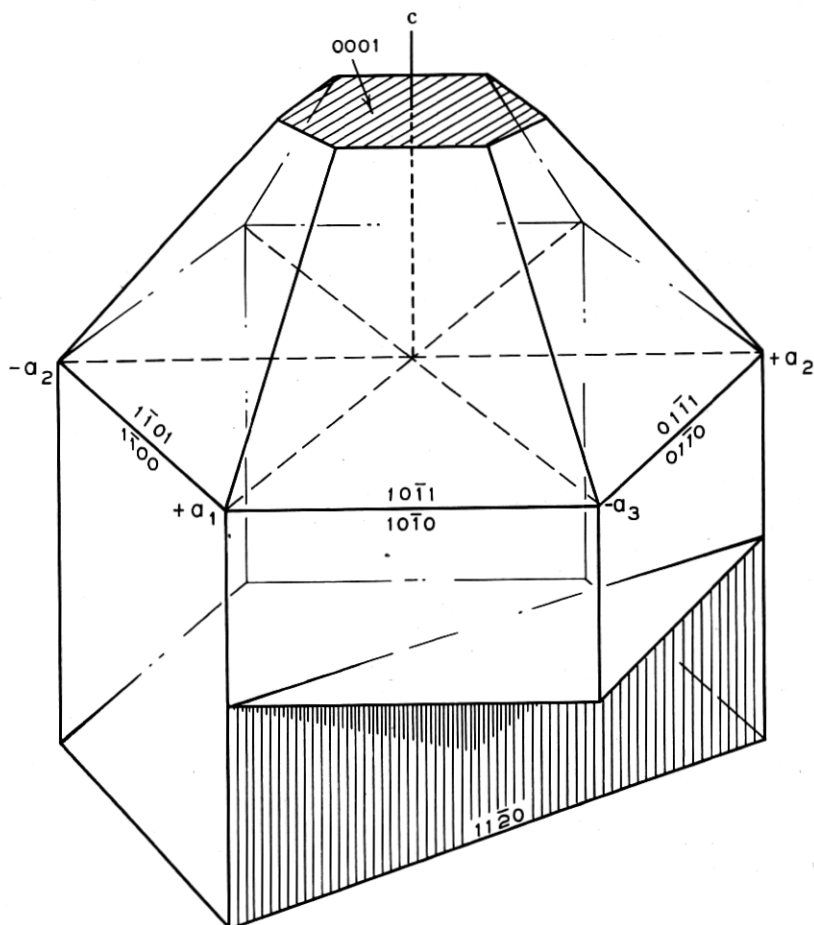

Fig. 3.5—Miller-Bravais indices of certain crystallographic planes in quartz

The use of indices instead of the actual intercepts simplifies the mathematics of crystallography in many ways. For example, the sum of the indices of two planes forms the indices of an intermediate plane that is parallel to the line of intersection of the first two. Thus the sum of $(00\cdot1)$, the atomic plane which is normal to c, and $(10\cdot0)$, prism face, is $(10\cdot1)$, the indices of the cap face directly above the $(10\cdot0)$ prism face.

Further, in the equation $n\lambda = 2d \sin \theta$, the interplanar spacing d of the atomic planes $(hk \cdot \ell)$ is

$$d_{hk \cdot \ell} = \frac{a_0}{\sqrt{\frac{4}{3}(h^2 + hk + k^2) + \frac{\ell^2}{(c/a)^2}}} \qquad (3.2)$$

Where the axial ratio c/a is 1.09997 in quartz and a_0, the distance between atoms along the a axis, is 4.903Å.[4]

The n order reflection from the $hk \cdot \ell$ plane is sometimes spoken of as the reflection from the $n(hk \cdot \ell)$ plane as in Table I and Fig. 3.8 where, for example, the second order reflection from the $01 \cdot 1$ plane is listed as the reflection from the $02 \cdot 2$ plane. The justification for this notation is that the $02 \cdot 2$ planes would have a d value half that of the $01 \cdot 1$ planes (since the indices are reciprocals of the axial intercepts) and the first order reflection from such planes would have the same θ value as the second order reflections from the $01 \cdot 1$ planes i.e., if

$$2\lambda = 2d \sin \theta$$

then

$$\lambda = 2 \frac{d}{2} \sin \theta$$

Among manufacturers of piezoelectric units a rectangular coordinate system is used in place of the hexagonal one described above, but the indices as derived from the hexagonal axes are retained. One of the a axes is chosen as the X axis. The three a axes are identical (the vertical axis of quartz is an axis of 3-fold symmetry) and therefore any one of the a axes may be chosen as X. The c axis is called the Z axis and a Y axis, normal to X and Z is so chosen as to form a right-handed coordinate system for right-handed quartz or a left-handed coordinate system for left-handed quartz as described in Chapter II.

The indices of the cap faces of right and left-handed quartz crystals (as viewed from above) are given in Fig. 3.6 which should be compared with Fig. 2.4 and 2.6 of Chapter II. (Note that Z in Fig. 3.6 refers to the Z axis whereas z in Fig. 2.6 refers to the z faces or minor cap faces). The cap faces directly beneath those illustrated (i.e., on the other end of a doubly terminated crystal) would have the same indices except that the final digit would be negative since they cut the negative end of the Z (or c) axis. Thus $(0\bar{1} \cdot \bar{1})$ is beneath $(0\bar{1} \cdot 1)$ and parallel to $(01 \cdot 1)$.

Parallel crystal faces lie along the same set of atomic planes. Thus $(01 \cdot 1)$ and $(0\bar{1} \cdot \bar{1})$ represent the same atomic plane. Further, since the

[4] Wyckoff, W. G., "The Structure of Crystals," The Chemical Catalogue Co. (1931).

vertical axis is an axis of 3-fold symmetry, the major cap-face planes $(01 \cdot 1)$, $(\bar{1}0 \cdot 1)$ and $(1\bar{1} \cdot 1)$ have the same properties and this applies also of course to the parallel crystal faces (same atomic planes) which are, respectively, $(0\bar{1} \cdot \bar{1})$, $(10 \cdot \bar{1})$, and $(\bar{1}1 \cdot \bar{1})$. Thus we can choose any one of these symbols to represent this type of plane when speaking of such properties as distance between atomic planes, θ angle, or angle between atomic planes and the Z axis. The symbol that will be used in this paper for this type of plane is $(01 \cdot 1)$.

Similar considerations apply to the set of minor cap faces for which the symbol used here is $(01 \cdot \bar{1})$ which is chosen instead of $(0\bar{1} \cdot 1)$ because it simplifies tabulation, as in Table I and Fig. 3.8.

The indices $(hk \cdot \ell)$ of all faces or atomic planes with the same X-ray properties as those for any given plane may be derived as follows: First replace the omitted digit i in the indices of the given plane, which may be
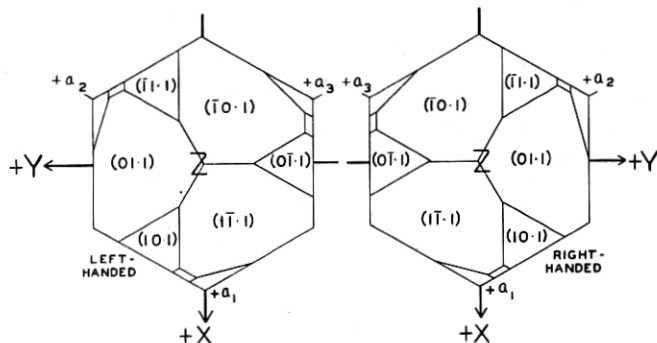

Fig. 3.6—Indices of cap-faces of right- and left-handed quartz crystals

determined from the equation $i = -(h + k)$. Rotary permutation of the first three indices is then permitted, as $a\,b\,\bar{c}\,\ell$, $\bar{c}\,a\,b\,\ell$, $b\,\bar{c}\,a\,\ell$.

The other three of the six equivalent faces are found by interchanging any adjacent two of the first three indices and also changing the sign of ℓ, as $a\,b\,\bar{c}\,\ell$ becomes $a\,\bar{c}\,b\,\bar{\ell}$ or $b\,a\,\bar{c}\,\bar{\ell}$. For example:

$$1\ 2\ \bar{3}\ 4 \qquad \bar{3}\ 1\ 2\ 4 \qquad 2\ \bar{3}\ 1\ 4$$

$$1\ \bar{3}\ 2\ \bar{4} \qquad 2\ 1\ \bar{3}\ \bar{4} \qquad \bar{3}\ 2\ 1\ \bar{4}$$

We could consider the $2\ 1\ \bar{3}\ \bar{4}$ as derived from cyclic permutation of $1\ \bar{3}\ 2\ \bar{4}$, and $\bar{3}\ 2\ 1\ \bar{4}$ as similarly derived from $2\ 1\ \bar{3}\ \bar{4}$ instead of by interchange of the sets above them.

The six symbols derived from the above by changing the sign of only the final digit refer to faces whose θ X-ray angle is the same as that of the above planes but whose intensity of reflection may be different.

TABLE I

QUARTZ X-RAY REFLECTION ANGLES

The Planes of Quartz from which the $Cu\ K_\alpha$ will reflect

θ = Bragg angle of X-ray reflection for $Cu\ K_\alpha$ radiation
γ = Angle that the plane normal N makes with the Z axis
ϵ = The angle between X and the plane containing the normal and Z, measured in the XY plane
I = Relative intensity of reflection

$hk \cdot \ell$	θ	γ	ϵ	I for $\ell+$	I for $\ell-$	$hk \cdot \ell$	θ	γ	ϵ
00·3	25°20'	0°	90°	.50	.50	11·0	18°17'	90°	60°
00·6	58°49'	0°	"		.38	11·1	20° 9'	65°33'	60°
01·0	10°26'	90°	"	24.	24.	11·2	25° 5'	47°44'	60°
01·1	13°20'	51°47'	"	100.	75.	11·3	32° 3'	36°15'	60°
01·2	19°45'	32°25'	"	28.	85.	11·4	40°37'	28°49'	60°
01·3	27°41'	22°57'	"	45.	2.7	11·5	51°10'	23°45'	60°
01·4	36°45'	17°37'	"			11·6	65°41'	20° 8'	60°
01·5	47°21'	14°15'	"			12·0	28°38'	90°	70°54'
01·6	60°59'	11°57'	"			12·1	29°59'	73°26'	70°54'
02·0	21°14'	90°	"	28.	28.	12·2	33°53'	59°14'	70°54'
02·1	22°55'	68°31'	"			12·3	39°58'	48°15'	70°54'
02·2	27°27'	51°47'	"	26.	26.	12·4	46°44'	40° 2'	70°54'
02·3	34° 5'	40°16'	"	32.	63.	12·5	58°59'	33°54'	70°54'
02·4	42°30'	32°25'	"			12·6	78°41'	29°15'	70°54'
02·5	53° 6'	26°56'	"			13·0	40°46'	90°	76° 6'
02·6	68°17'	22°57'	"			13·1	41°56'	77°41'	76° 6'
03·0	32°54'	90°	"			13·2	45°35'	66°25'	76° 6'
03·1	34°10'	75°18'	"	40.	1.4	13·3	51°20'	56°46'	76° 6'
03·2	37°51'	62°18'	"	19.	27.	13·4	60° 7'	48°52'	76° 6'
03·3	43°45'	51°47'	"			13·5	75°11'	42°29'	76° 6'
03·4	51°58'	43°37'	"	6.3	3.2	14·0	56° 6'	90°	79° 6'
03·5	63°41'	37°19'	"			14·1	57°22'	80°15'	79° 6'
04·0	46°25'	90°	"			14·2	61°21'	71° 2'	79° 6'
04·1	47°35'	78°52'	"			14·3	69° 1'	62°44'	79° 6'
04·2	51° 8'	68°31'	"			22·0	38°51'	90°	60°
04·3	57°17'	59°26'	"			22·1	40° 3'	77°12'	60°
04·4	67°13'	51°47'	"	12.	0.7	22·2	43°34'	65°34'	60°
05·0	64°54'	90°	"			22·3	47°54'	55°43'	60°
05·1	66°27'	81° 3'	"			22·4	57°59'	47°44'	60°
05·2	71°42'	72°31'	"	.25	23.	22·5	71°45'	41°21'	60°
						23·0	52° 8'	90°	66°35'
						23·1	53°20'	76°40'	66°35'
						23·2	57° 4'	70° 8'	66°35'
						23·3	63°53'	61°33'	66°35'
						23·4	76°52'	54° 9'	66°35'
						24·0	73°24'	90°	70°54'
						24·1	75°40'	81°32'	70°54'
						24·2	88°54'	73°26'	70°54'
						33·0	70°15'	90°	60°
						33·1	72° 8'	81°23'	60°
						33·2	79°32'	73° 9'	60°
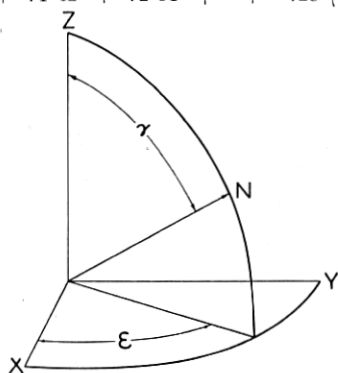
The axes (and therefore the indices) given for left-handed quartz are the mirror image of those for right-handed quartz so that similar faces have the same indices in both and the signs of the crystallographic axes are consistent with those of the electrical and physical properties in both. (Crystallographers commonly use the same set of axes for both right and left-handed quartz.) Table I gives the orientation and X-ray properties
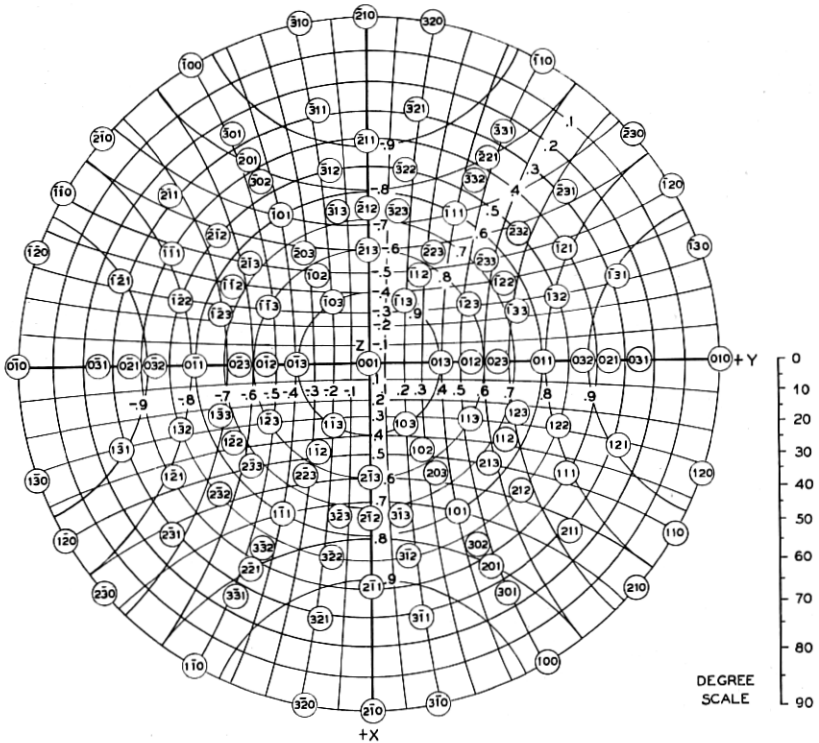

Fig. 3.7—Stereographic projection of normals to the atomic planes in quartz

of the atomic planes in quartz that will reflect X-rays with appreciable intensity.

The θ, γ, and ϵ values have been calculated from the dimensions of the quartz crystal lattice as determined by X-ray investigation. The intensity values were determined experimentally and are only approximate. Figure 3.8 shows the geometrical disposition around the X axis of those planes that are parallel to the X axis (column 1, Table I) together with the X-ray data for these planes and the disposition around the X axis of the common single-rotation cuts. Figure 3.7 is a stereographic projection of the normals of
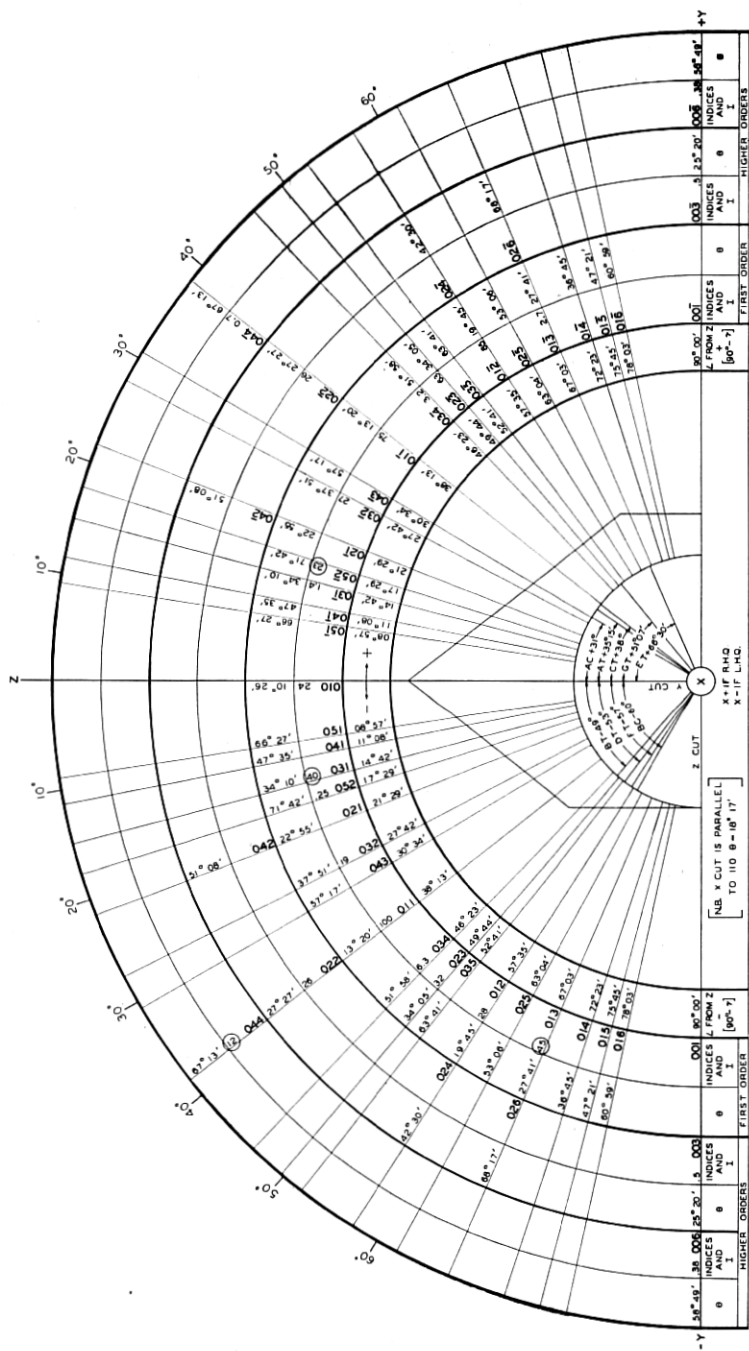
Fig. 3.8—Atomic-planes and common plate-cuts parallel to the X axis

the more important atomic planes of quartz, identified by their indices. Its usefulness in quartz work will be pointed out in Section 3.7B.

In many cases the reflecting power of an atomic plane differs from that of the symmetrical plane on the other side of the Z axis. (See, for example $01 \cdot 1$ and $01 \cdot \bar{1}$). When this difference is very great as with $04 \cdot 4$ and $04 \cdot \bar{4}$ the planes are useful in determining whether a plate is cut at a positive or negative angle from the Z axis. The intensities of planes that have been found useful for this purpose are circled in Fig. 3.8.

3.6 X-Ray Goniometry

Since the angles θ and the intensities I are different for different planes we can use them to identify these planes, that is, to orient the crystal by measuring angles between recognized atomic planes and plate surfaces.

Figure 3.9 is a diagrammatic representation of an X-ray goniometer where

T is the tube target shown with its intensity pattern,

SS are slits that pass only a narrow beam,
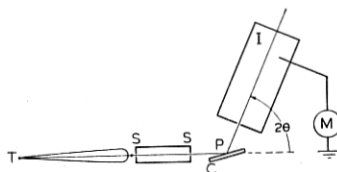
C is the crystal,

Fig. 3.9—Simplified diagram of X-ray goniometry

I is the ionization chamber

M is the meter that measures the ionization current.

The ionization chamber is placed at an angle 2θ to the incident beam, where θ is the Bragg angle for the atomic plane being used, and is not moved while reflections are being taken from that atomic plane. If C is then rocked about the vertical axis P (normal to the plane of the paper) the ionization chamber registers an electric current when an atomic plane is at the proper angle for reflection.

(a) Atomic plane parallel to plate-face.

Let us examine a simple case, that for which the existing face is parallel to an atomic plane (Fig. 3.10). The crystal is held against the reference points by a coil spring. The crystal holder is free to rotate about the vertical axis P (with respect to the X-rays) and the angle of rotation is read on the graduated scale. If the entering angle (the angle between the entering beam and the plate-face) is one that satisfies the equation $n\lambda = 2d \sin \theta$, we will have a reflected ray which is at a leaving angle of θ. Also the reflected ray is always deviated from the line of the original

ray by the angle 2θ. If, when the crystal gives a reflected ray, it is rotated in its own plane on the reference points the reflection is unchanged.

(b) Atomic plane intersecting plate-face in a line normal to *the plane of the instrument*.[5]

If in the above case the rotation of the crystal in its own plane does cause a change of ionization current the surface is not parallel to the atomic plane. Figure 3.11 illustrates a case in which the surface is at an angle δ to an atomic plane, this intersection lying normal to the plane of the paper in Fig. 3.11. In order that the angle between the X-ray beam and the atomic plane shall be θ, the angle between the beam and the plate-face must now be $\theta - \delta$ on one side and $\theta + \delta$ on the other. The deviation angle 2θ is the sum of these two. Therefore for atomic planes intersecting
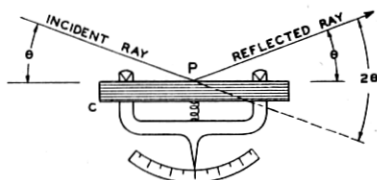

Fig. 3.10—Goniometry case (a): Atomic plane parallel to plate-face
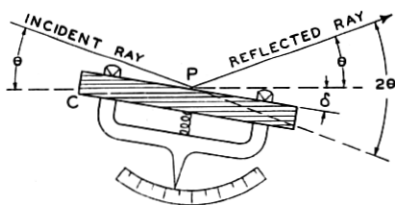

Fig. 3.11—Goniometry case (b): Atomic plane intersecting plate-face in a line normal to "the plane of the instrument"

the plate-face in a line normal to the plane of the instrument the ionization chamber is set at 2θ regardless of the angle between the atomic plane and the surface of the plate.

In Fig. 3.11 the entering angle is shown as $\theta - \delta$, the leaving angle as $\theta + \delta$. If the crystal is now rotated through an angle of $180°$ in its own plane the entering angle will have to be set at $\theta + \delta$ and the leaving angle will become $\theta - \delta$. The crystal holder will have been moved through an angle 2δ. Thus δ can be found by observing the angle through which the crystal holder must be rotated in order to achieve X-ray reflection from the same atomic plane when the crystal is rotated $180°$ in its own plane.

[5] The *plane of the instrument* is a plane normal to the axis of rotation of the instrument and containing the incident ray.

It should be noted that δ should not exceed θ. If δ exceeds θ, it is necessary for the X-ray beam to pass through a great thickness of quartz which would so weaken the beam that the reflected rays could not be detected. Further, even when δ does not exceed θ there is still a variation of the reflected ray intensity with the angle between the entering ray and the plate-face, which may be expressed as follows:[6]

$$I_r = I_R \frac{\sin g_\ell}{\sin g_\ell + \sin g_e}$$

where

I_r = The intensity of the reflected ray

g_e = The "entering angle" = The angle between the entering ray and the plate face

g_ℓ = The "leaving angle" (In Fig. 3.11 $g_e = \theta - \delta$; $g_\ell = \theta + \delta = 2\theta - g_e$)

I_R = The maximum obtainable reflected intensity (i.e., when $g_e = 0$ and $g_\ell = 2\theta$).

or

$$I_r = I_R \frac{\sin (2\theta - g_e)}{\sin (2\theta - g_e) + \sin g_e}$$

The curves in Fig. 3.12 show the variation of I_r/I_R with $g_e/2\theta$ for various values of 2θ. It is seen that for plates as described above and figured in 3.11 a stronger reflection is obtained when $g_e = \theta - \delta$ than when $g_e = \theta + \delta$ (i.e., after 180° rotation in the plane of the plate-face). In other words the smaller g_e angle gives the larger intensity of reflection. It might be added, however, that in practice it may not be possible to obtain the maximum values of reflection intensity due to the fact that, as g_e approaches zero, the width of the reflected beam may exceed the width of the ionization chamber. (See inset, Fig. 3.12.)

Quartz plates containing an X axis are checked as described above (case b) for rotation around the X axis. For this check they are oriented as shown in Fig. 3.13, case b with the X axis parallel to the axis of the instrument. The stippled face in Fig. 3.13 represents the so-called "reference bevel", the remnant of the surface of the Z section, and is therefore normal to the Z axis.

(c) Atomic plane intersecting plate-face in a line parallel to the plane of the instrument.

The case described above and illustrated in Fig. 3.11 was the case of single rotation plates (AT, BT and others shown in Fig. 3.8) being corrected about the X axis by the use of atomic planes parallel to the X axis. When

[6] Debye, P. and Menke, H., "Untersuchung der Molekularen Ordnung in Flüssigkeiten mit Röntgenstrahlung," Ergeb d. Techn. Röntgenkunde, B.2, P. 16; Leipzig (1931).

such a plate is being rotated about the X axis as in Fig. 3.11 the normal to the atomic plane lies in the plane of the instrument. We can also use
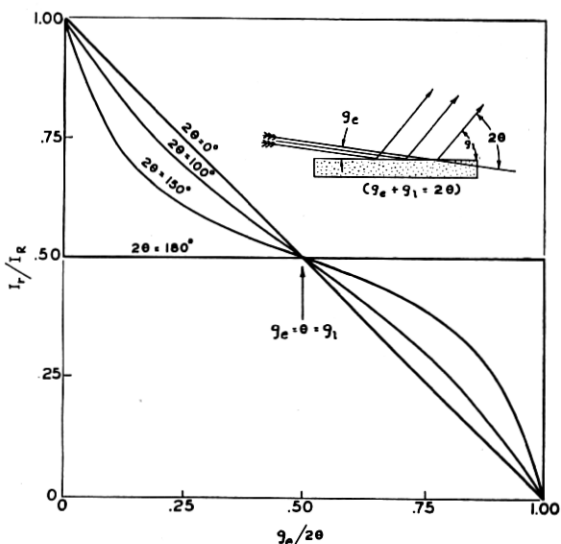

Fig. 3.12—Variation of intensity of reflected ray with entering angle g_e and the Bragg angle, θ
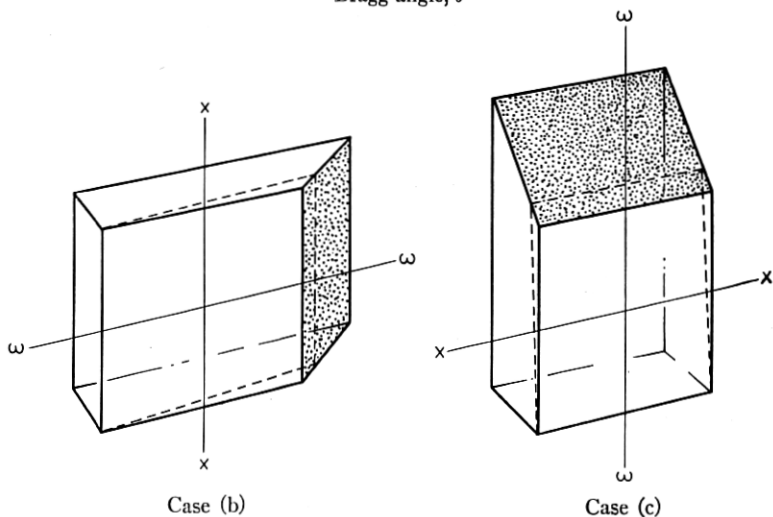

Case (b)   Case (c)

Fig. 3.13—Position of quartz plate relative to vertical axis of instrument in case (b) and case (c). (Stippled face is reference bevel, normal to Z axis.)   In the absence of a reference bevel the X direction can be determined with polarized light. (See Chapter II, p. 246.)

the same atomic plane to correct the orientation of this plate about the axis ww normal to the X axis (Fig. 3.13 and Fig. 3.14). Here, since the

atomic planes are at an angle δ to the axis of the instrument, the reflected ray will not lie in the plane of the instrument. If, however, the vertical angle β, between the reflected ray and the plane of the instrument is small, the reflected ray may still enter the ionization chamber. The maximum angle β which is permissible, is not the same for all instruments, but depends on the vertical length of the slit. In most cases it is about 5°.

The proper angular settings of the instrument are no longer θ for the slab and 2θ for the ionization chamber but are the orthogonal projection of these angles onto the plane of the instrument; g and $g + g'$, respectively.
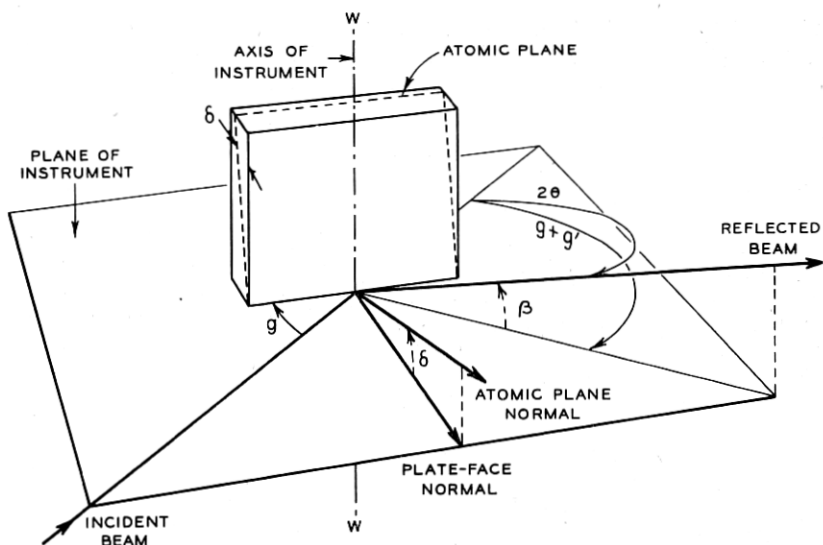

Fig. 3.14—Goniometry case (c): Atomic plane intersecting plate-face in a line parallel to "the plane of the instrument"

Formulae are given below for β, g, and $g + g'$ in terms of the Bragg angle θ and the angle between the atomic planes and the surface of the quartz plate, δ.

$$\sin \beta = 2 \sin \theta \sin \delta \tag{3.3}$$

$$\cos (g + g') = \frac{\cos 2\theta}{\cos \beta} \tag{3.4}$$

$$\sin g = \frac{\sin \theta}{\cos \delta} \tag{3.5}$$

The relations between β, $g + g'$, g, θ, and δ are shown by the curves in Fig. 3.15.

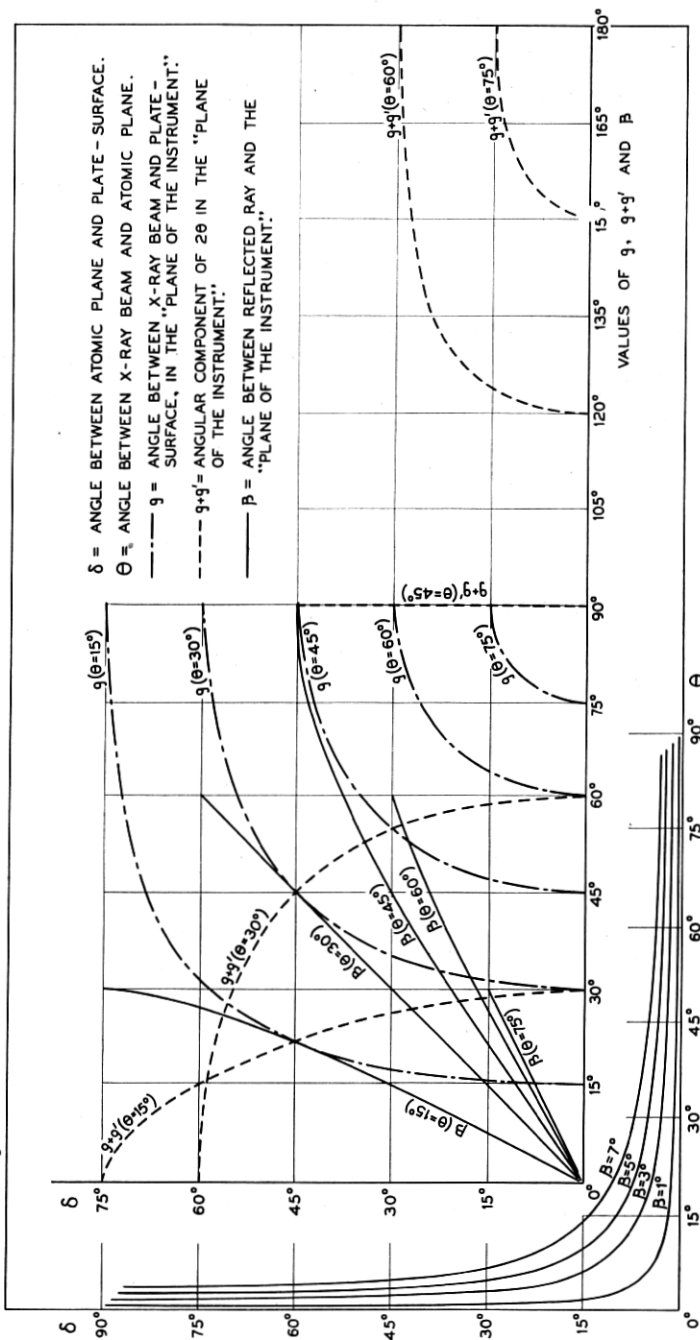As an example, suppose that a BT plate is to be corrected about the

Fig. 3.15—Variation of β, g and $g + g'$ with δ and θ

ww axis using the atomic plane $(01\cdot1)$. (In actual practice the closer plane $(02\cdot3)$ is used.) Here:

$$\delta = 49° - 38°13' = 10°47', \qquad \theta = 13°20', \qquad 2\theta = 26°40'$$

From which $\qquad \beta = 4°57', \qquad g = 13°35', \qquad g + g' = 26°34'$

Thus if the plate is correctly cut the reflected ray will enter an ionization chamber whose slit is long enough to receive rays which make an angle of 5° with the plane of the instrument. However, if the plate were in error by more than 6' around the XX axis (Fig. 3.13) β would excede 5° and the center of the reflected beam would not enter the ionization chamber. In actual practice, therefore, the length of the ionization chamber slit should be enough greater than that required for the calculated β to admit reflections from erroneously cut plates.

Since the difference between $g + g'$ and 2θ is only 6' and since the width of the ionization chamber slit is usually great enough to accept reflected beams over a range of several times 6', no correction of the ionization chamber position may be necessary in this case.

On the other hand, the orientation of the plate with respect to the incident beam is highly critical and since g differs from θ by 15' this correction in the orientation of the quartz plate must be made.

Discussion of the general case (d), in which the intersection of the atomic plane and the plate face is neither normal nor parallel to the plane of the instrument, will be found in Section 3.9.

3.7 CHOICE OF AN ATOMIC PLANE FOR CHECKING THE ORIENTATION OF ANY GIVEN FACE

If the plate-face to be checked does not lie parallel to an atomic plane, the nearest usable atomic plane must be found and the orientation of this atomic plane in the plate must be determined. The procedure for these two steps is outlined in this and the following sections.

The problem of the choice of an atomic plane for checking the orientation of one of the faces of a given plate has two parts:

(A) Determination of the orientation of that face with respect to the X, Y and Z axes of the mother crystal and (B) discovery of the atomic plane whose orientation and X-ray properties are most suitable for use with that face.

(A) *Determination of the orientation of the face with respect to the* X, Y *and* Z *axes of the mother crystal.*

The orientation of the plate is commonly given in terms of the A_1, A_2 and A_3 angular rotations as described and illustrated in Section 2.4 of Chapter II. Briefly, a basal section of crystal is placed initially with its Z axis vertical and its $+X$ axis toward the operator of a horizontal axis saw,

the saw blade being parallel to X and Z.   The crystal is then turned through angle A_1 clockwise as seen from above about a vertical axis, then through angle A_2 counterclockwise about the original direction of the X axis.   A slab is cut of thickness t and this slab layed down by rotating it 90° clockwise about the original X axis direction.   It is then rotated through angle A_3 clockwise about a vertical axis and two cuts are made, separated by a width w (length ℓ being cut last).   The rotation through the A_3 angle begins with the linear edge of the reference level of the slab lying parallel to the saw.

The components of the plate edges P_1, P_2, P_3 (length, thickness, and width, respectively) on the X, Y, and Z coordinates after rotation through the angle A_1 are given by the following matrix (See Section 5 of "The Mathematics of the Physical Properties of Crystals" by W. L. Bond, *Bell System Technical Journal*, Volume XXII, No. 1):

$$r' = \begin{array}{cccc} X\ \text{component} & Y\ \text{component} & Z\ \text{component} & \\ \left(\begin{matrix} \cos A_1 & \sin A_1 & 0 \\ -\sin A_1 & \cos A_1 & 0 \\ 0 & 0 & 1 \end{matrix} \right. & & \left. \begin{matrix} \\ \\ \end{matrix} \right) & \begin{matrix} \text{of } P_1 \\ \text{of } P_2 \\ \text{of } P_3 \end{matrix} \end{array} \dots \dots (3.6)$$

The components of the plate edges on the X, Y, Z coordinates after rotation through angles A_1 and A_2 are:

$$r'' = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos A_2 & -\sin A_2 \\ 0 & \sin A_2 & \cos A_2 \end{pmatrix} \begin{pmatrix} \cos A_1 & \sin A_1 & 0 \\ -\sin A_1 & \cos A_1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

$$= \begin{array}{cccc} X\ \text{component} & Y\ \text{component} & Z\ \text{component} & \\ \left(\begin{matrix} \cos A_1 & \sin A_1 & 0 \\ -\sin A_1 \cos A_2 & \cos A_1 \cos A_2 & -\sin A_2 \\ -\sin A_1 \sin A_2 & \cos A_1 \sin A_2 & \cos A_2 \end{matrix} \right) & \begin{matrix} \text{of } P_1 \\ \text{of } P_2 \\ \text{of } P_3 \end{matrix} \end{array} (3.7)$$

The components of the plate edges on the X, Y, Z coordinates after rotation through angles A_1, A_2 and A_3 are:

$$r''' = \begin{pmatrix} \cos A_3 & 0 & \sin A_3 \\ 0 & 1 & 0 \\ -\sin A_3 & 0 & \cos A_3 \end{pmatrix} \begin{pmatrix} \cos A_1 & \sin A_1 & 0 \\ -\sin A_1 \cos A_2 & \cos A_1 \cos A_2 & -\sin A_2 \\ -\sin A_1 \sin A_2 & \cos A_1 \sin A_2 & \cos A_2 \end{pmatrix}$$

$$= \begin{array}{cccc} X\ \text{component} & Y\ \text{component} & Z\ \text{component} & \\ \left(\begin{matrix} \cos A_1 \cos A_3 \\ -\sin A_1 \sin A_2 \sin A_3 \end{matrix} \right. & \begin{matrix} \sin A_1 \cos A_3 \\ +\cos A_1 \sin A_2 \sin A_3 \end{matrix} & \begin{matrix} \cos A_2 \sin A_3 \\ \ \end{matrix} & \begin{matrix} \text{of } P_1 \\ \ \end{matrix} \\ -\sin A_1 \cos A_2 & \cos A_1 \cos A_2 & -\sin A_2 & \text{of } P_2 \\ \left. \begin{matrix} -\cos A_1 \sin A_3 \\ -\sin A_1 \sin A_2 \cos A_3 \end{matrix} \right. & \begin{matrix} -\sin A_1 \sin A_3 \\ +\cos A_1 \sin A_2 \cos A_3 \end{matrix} & \left. \begin{matrix} \cos A_2 \cos A_3 \\ \ \end{matrix} \right) & \begin{matrix} \text{of } P_3 \\ \ \end{matrix} \end{array} \dots (3.8)$$

Since the edge P_2 (thickness direction) is the normal to the major face of the plate, its X, Y, Z components (second horizontal row) give the orientation of the major face of the plate in terms of X, Y, Z axes of the mother crystal.

For example, suppose the orientation of the major face of an NT plate is desired in terms of the X, Y, Z axes. The shop rotation angles for an NT plate are

$$A_1 = 99°25'$$
$$A_2 = 49°25'$$
$$A_3 = -12°20'$$

Substituting these values in the above matrix gives

$$r''' = \begin{pmatrix} 0 & .99027 & -.13917 \\ -.64279 & -.10662 & -.75852 \\ -.76604 & .08946 & .63653 \end{pmatrix} \dots\dots (3.9)$$

the second row of which gives the components (direction cosines) of the unit normal to the plate surface on the X, Y, Z axes:

$$X = .64279$$
$$Y = .10662$$
$$Z = .75852$$

(B) Discovery of the plane whose orientation and X-ray properties are most suitable for use with the face to be checked.

The first requisite for the atomic plane to be used is that it shall make as small an angle δ as possible with the face to be checked. This is desirable because (1) δ must be smaller than θ; (2) if δ is small the same plane can be used for correction around P_1 as around P_3 since the β angle will be small. If δ is very small the difference between g and θ may be within the required limits of error so that the θ value may be used without correction; (3) if δ is large, the difference between g_e and g_ℓ will be large and if the plate is placed in the goniometer in such a way that g_e is the larger angle the reflected ray may be too weak to register on the ionization chamber meter.

By plotting the stereographic projection of the normal to the plate face on Fig. 3.7 the most promising planes may be found quickly. On this chart each concentric circle connects all points with the same Z direction-cosine; each arc that crosses the X axis connects all points with the same X direction-cosine; each arc that crosses the Y axis connects all points with the same Y direction-cosine.

For example, the normal to the major face of the NT cut is plotted as follows:

.64 units are marked off in a positive direction (down) on the X axis (to

a point between $(2\bar{1}\cdot3)$ and $(2\bar{1}\cdot2)$. Then, in the direction of the east-west arcs, .1 unit is marked off to the right of the X axis. The point so located is found to be about half way between the .7 and .8 concentric circles, that is, to have a Z component of about .75, as calculated above. It lies about half way between atomic plane normals $(2\bar{1}\cdot3)$ and $(3\bar{1}\cdot3)$.

The degree scale given in Fig. 3.7 may be used to determine roughly the angle between any two points on the diagram. The scale is non-uniform and for any particular region of the diagram that portion of the scale should be used that falls on that region when the zero mark on the scale is placed at the center of the diagram.
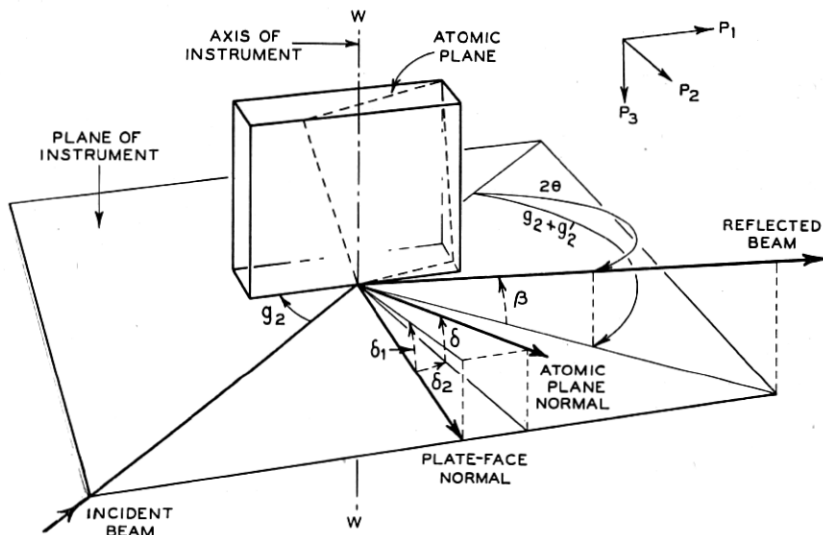
Fig. 3.16—Goniometry case (d): Atomic plane intersecting plate-face in a line which is neither normal nor parallel to "the plane of the instrument" (general case).

In the present example the part of the scale to be used is around the 40° mark and the plate face is found to make angles of less than 10° with both $(2\bar{1}\cdot3)$ and $(3\bar{1}\cdot3)$.

In general the atomic planes with the smaller indices are likely to give the stronger reflections. Therefore the better plane to try first is $(2\bar{1}\cdot3)$. The indices of this plane are not shown in this form in Table I, but by the method described in Section 3.5 it may be seen that a plane with indices $11\cdot3$ would have the same θ value for X-ray reflection. From Table I we see that θ for atomic plane $11\cdot3$ is 32° 03′.

In order to determine the angles g and $g + g'$ (Fig. 3.16) the orientation of the atomic plane with respect to the plate edges must be found.

3.8 Determination of the Orientation of an Atomic Plane with Respect to the Plate-Edges, Given its Miller-Bravais Indices

Since the indices $(hk \cdot \ell)$ are the reciprocals of the intercepts of the atomic plane on the quartz crystallographic axes (See Section 3.5) of which the vertical axial unit is 1.09997 times that of the others, the intercepts on axes which were all divided into the same length unit would be

$$\frac{1}{h}, \quad \frac{1}{k}, \quad \text{and} \quad \frac{1.1}{\ell}$$

(If 1.1 is substituted for 1.09997 the error introduced is never greater than one minute.)

The intercepts on the orthogonal axes may be derived as indicated in Fig. 3.17. They are:

$$\frac{1}{h}, \quad \frac{\sqrt{3}}{h + 2k}, \quad \frac{1.1}{\ell}$$

These intercepts may be taken as the lengths of 3 vectors whose components on the X, Y and Z axes are as follows:

$$\iota_1(X \text{ axis vector}) = \begin{pmatrix} 1/h \\ 0 \\ 0 \end{pmatrix} \dots\dots\dots\dots (3.10)$$

$$\iota_2(Y \text{ axis vector}) = \begin{pmatrix} 0 \\ \dfrac{\sqrt{3}}{h + 2k} \\ 0 \end{pmatrix} \dots\dots\dots (3.11)$$

$$i_3(Z \text{ axis vector}) = \begin{pmatrix} 0 \\ 0 \\ 1.1/\ell \end{pmatrix} \dots\dots\dots (3.12)$$

Now the two vectors $(i_3 - i_1)$ and $(i_3 - i_2)$ lie in the plane $(hk \cdot \ell)$ (See Fig. 3.18), and therefore their vector product is the vector normal to the plane $hk \cdot \ell$
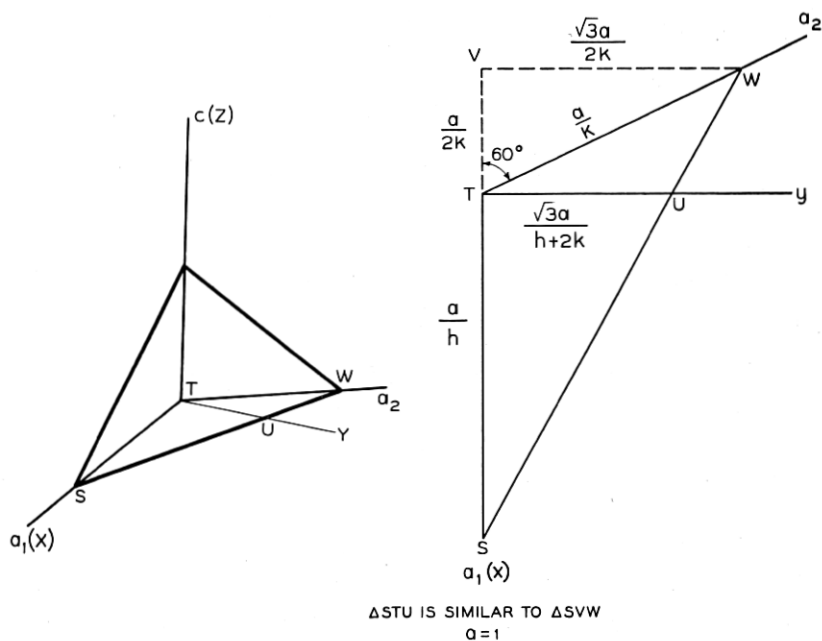
ΔSTU IS SIMILAR TO ΔSVW

a = 1

Fig. 3.17—Derivation of intercepts on orthogonal axes from intercepts on hexagonal axes
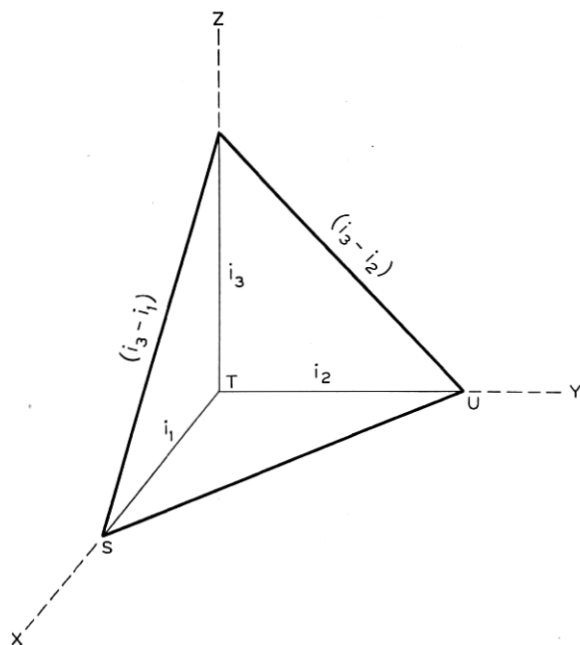

Fig. 3.18—Orientation of vectors (i_3-i_1) and (i_3-i_2) with respect to orthogonal axes

That is,

$\bar{N}$(the vector normal to the atomic plane $hk \cdot \ell$)

$$= \left[\begin{pmatrix} 0 \\ 0 \\ 1.1/\ell \end{pmatrix} - \begin{pmatrix} 1/h \\ 0 \\ 0 \end{pmatrix} \right] \times \left[\begin{pmatrix} 0 \\ 0 \\ 1.1/\ell \end{pmatrix} - \begin{pmatrix} 0 \\ \dfrac{\sqrt{3}}{h+2k} \\ 0 \end{pmatrix} \right]$$

$$= \begin{pmatrix} -1/h \\ 0 \\ 1.1/\ell \end{pmatrix} \times \begin{pmatrix} 0 \\ -\dfrac{\sqrt{3}}{h+2k} \\ 1.1/\ell \end{pmatrix}$$

$$= \begin{pmatrix} 0 & -1.1/\ell & 0 \\ 1.1/\ell & 0 & 1/h \\ 0 & -1/h & 0 \end{pmatrix} \begin{pmatrix} 0 \\ -\dfrac{\sqrt{3}}{h+2k} \\ 1.1/\ell \end{pmatrix}$$

$$= \begin{pmatrix} \dfrac{1.1\sqrt{3}}{\ell(h+2k)} \\ 1.1/h\ell \\ \dfrac{\sqrt{3}}{h(h+2k)} \end{pmatrix} = \begin{pmatrix} h \\ h+2k \\ \dfrac{\sqrt{3}}{\ell/1.1} \end{pmatrix} \dots \dots \dots \dots \dots \quad (3.13)$$

The unit vector $N = \dfrac{1}{\sqrt{S}} \bar{N}$ $\dots \dots \dots \dots \dots \dots (3.14)$

where S is the sum of the squares of the three terms of $\bar{N}$. For example, for the $(01 \cdot 1)$ plane ($h = 0$, $k = 1$, $\ell = 1$), we find from equation (3.13) that

$$\bar{N} = \begin{pmatrix} 0 \\ 2/\sqrt{3} \\ 1/1.1 \end{pmatrix} \dots \dots \dots \dots \dots (3.15)$$

whence $S = 4/3 + \dfrac{1}{1.21} = 2.159$ $\dots \dots \dots \dots \dots \dots (3.16)$

and the unit vector normal $N = \begin{pmatrix} 0 \\ .7857 \\ .6186 \end{pmatrix} \dots \dots \dots \dots \dots (3.17)$

which means that N is perpendicular to the X axis, makes an angle with Y whose cosine is .7857 ($=38° \, 13'$) and an angle with Z whose cosine is .6186 ($=51° \, 47'$).

For the atomic plane $(2\bar{1}\cdot3)$ we find from equation (3.13) that

$$\overline{N} = \begin{pmatrix} 2 \\ 0 \\ 3/1.1 \end{pmatrix} \quad \dots \dots \dots \dots \dots \dots (3.18)$$

Whence $S = 4 + 9/1.21 = 11.438 \dots \dots \dots \dots (3.19)$

and, from equation (3.14) the unit vector normal to $(2\bar{1}\cdot3)$

$$N = \begin{pmatrix} .5914 \\ 0 \\ .8064 \end{pmatrix} \quad \dots \dots \dots \dots \dots \dots (3.20)$$

We have now determined the orientation of the atomic plane with respect to the orthogonal axes X, Y and Z. To determine its orientation with respect to the plate edges necessitates the construction of a matrix which expresses the components of the plate edges $P_1 P_2 P_3$ (length, thickness, and width respectively) in terms of the X, Y, Z axes. When the components of the unit normal to the atomic plane in terms of the orthogonal axes X, Y, Z are acted upon by this matrix they are converted to the components of the unit normal to the atomic plane in terms of the plate edges P_1, P_2, P_3. (For fuller discussion see Section 5 of "The Mathematics of the Physical Properties of Crystals" by Walter L. Bond, *Bell System Technical Journal*, Volume XXII, No. 1, pp. 1–72.) Equation (3.8) gives such a matrix.

To continue with the NT cut as an example, the product of the matrix given in equation (3.9) for the NT cut and the components of the unit vector for the atomic plane $(2\bar{1}\cdot3)$ given in equation (3.20) gives us the components of the unit normal to the atomic plane $(2\bar{1}\cdot3)$ in terms of the plate edges P_1, P_2, P_3 as axes. Thus,

$$N_{P_1 P_2 P_2} = \begin{pmatrix} 0 & .99027 & -.13917 \\ -.64279 & -.10662 & -.75852 \\ -.76604 & .08946 & .63653 \end{pmatrix} \begin{pmatrix} .5914 \\ 0 \\ .8064 \end{pmatrix}$$

$$= \begin{pmatrix} -.11223 \\ -.99180 \\ +.060293 \end{pmatrix} \dots (3.21)$$

That is, the components of the unit normal to the atomic plane in terms of the plate edges P_1, P_2, P_3 are:

$$N_1 = -.11223$$
$$N_2 = -.99180$$
$$N_3 = +.060293$$

When the orientation of the atomic plane with respect to the plate edge has thus been determined the next and final step is the determination of the angles between the incident X-rays and the plate-face.

3.9 DETERMINATION OF ANGLES BETWEEN X-RAYS AND THE FACES OF A FINISHED PLATE

Procedures for determining the angles g and $g + g'$ for certain particular positions of the atomic plane with respect to the plate edges were described in section 3.6 under (a), (b), and (c). For the general case (d) in which the intersection between the atomic plane and the plate-face is neither normal nor parallel to the plane of the instrument, the problem is best solved vectorially, as follows:

(d) Atomic plane intersecting plate-face in a line which is neither normal nor parallel to the plane of the instrument (general case). (Fig. 3.16)
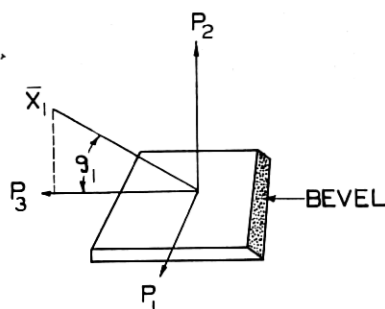
Fig. 3.19—Position 1 for a plate of general orientation (one in which no plate-edge is parallel to a crystallographic axis).

Let N_1, N_2, N_3 be the components of the unit normal $\overline{N}$ to the atomic plane in terms of the plate edges P_1, P_2, P_3, and X_1, X_2, X_3 the components of the unit vector $\overline{X}$ along the incident beam. Then

$$\sin \theta_{hk \cdot \ell} = X_1N_1 + X_2N_2 + X_3N_3 \qquad (3.22)$$

(the inner product of these two vectors, which is thus equal to the cosine of the angle between the incident ray and the normal to the atomic plane or $\cos (90 - \theta)$).

In matrix form this may also be written:

$$\overline{X}_c \, N_{hk \cdot \ell} = \sin \theta_{hk \cdot \ell} \qquad (3.23)$$

Where $\overline{X}_c$ is the matrix X_1, X_2, X_3 [7]

[7] See Bond, W. L. "The Mathematics of the Physical Properties of Crystals," *Bell Sys. Tech. Jour.*, Vol. XXII, No. 1.

From Fig. 3.19 we see that, for the beam entering as shown, the components of the unit vector $\bar{X}_1$ along the beam are

$$0$$
$$\sin g_1$$
$$\cos g_1$$

so that equation (3.22) becomes

$$\sin \theta_{hk \cdot \ell} = N_2 \sin g_1 + N_3 \cos g_1 \tag{3.24}$$

which has the solution

$$g_1 = \theta' - \delta_1' \tag{3.25}$$

where

$$\tan \delta_1' = \frac{N_3}{N_2} \tag{3.26}$$

and

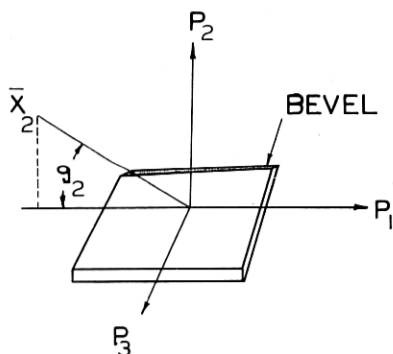$$\sin \theta' = \frac{\sin \theta}{\cos \sin^{-1} N_1} \tag{3.26'}$$

Fig. 3.20—Position 2 for a plate of general orientation

Again, with the plate rotated 90° around its normal (to position 2) so that the entering beam is in the position shown by the unit vector $\bar{X}_2$ in Fig. 3.20, the components of $\bar{X}_2$ are

$$-\cos g_2$$
$$\sin g_2$$
$$0$$

so that

$$\sin \theta = N_2 \sin g_2 - N_1 \cos g_2 \tag{3.27}$$

or

$$g_2 = \theta'' + \delta_2' \tag{3.28}$$

where $$\tan \delta_2' = \frac{N_1}{N_2} \tag{3.29}$$

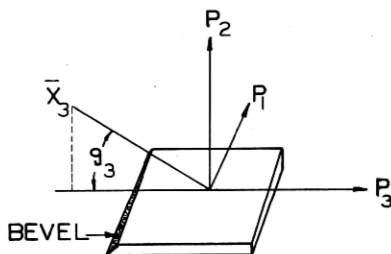and $$\sin \theta'' = \frac{\sin \theta}{\cos \sin^{-1} N_3} \tag{3.29'}$$

Fig. 3.21—Position 3 for a plate of general orientation

With the plate rotated 90° again (180° from position 1), so that the entering beam is in the position shown in Fig. 3.21, the components of the unit vector $\bar{X}_3$ along the incident beam are

$$0$$
$$\sin g_3$$
$$-\cos g_3$$

so that $$\sin \theta = N_2 \sin g_3 - N_3 \cos g_3 \tag{3.30}$$

or $$g_3 = \theta' + \delta_3' \tag{3.31}$$

where $$\tan \delta_3' = \frac{N_3}{N_2} \tag{3.32}$$

and $$\sin \theta' = \frac{\sin \theta}{\cos \sin^{-1} N_1} \tag{3.26'}$$

$$\delta_3' = \delta_1' \tag{3.33}$$

Finally, with the X-ray beam entering as shown by the unit vector $\bar{X}_4$ in Fig. 3.22 (270° from position 1) the components of $\bar{X}_4$ are

$$\cos g_4$$
$$\sin g_4$$
$$0$$

so that $$\sin \theta = N_2 \sin g_4 + N_1 \cos g_4 \tag{3.34}$$

or $$g_4 = \theta'' - \delta_4' \tag{3.35}$$

where
$$\tan \delta_4' = \frac{N_1}{N_2} \tag{3.36}$$

and
$$\sin \theta'' = \frac{\sin \theta}{\cos \sin^{-1} N_3} \tag{3.29'}$$

$$\delta_4' = \delta_2' \tag{3.37}$$

For example, suppose an NT finished crystal is to be checked by the $(2\bar{1}\cdot3)$ atomic plane as suggested in section 3.7B. The components of the unit normal to the atomic plane $(2\bar{1}\cdot3)$ in terms of the plate edges P_1, P_2, P_3 of an NT plate were found (at the end of section 3.8) to be

$$N_1 = -.11223$$
$$N_2 = -.99180$$
$$N_3 = .060293$$
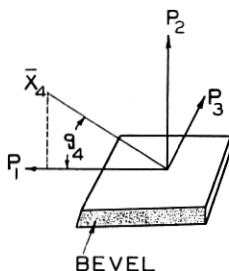
Fig. 3.22—Position 4 for a plate of general orientation

Substituting in equation (3.26):
$$\tan \delta_1' = \frac{.060293}{-.99180}$$

$$\delta_1' = -3°28.7'$$

and in equation (3.26')
$$\sin \theta' = \frac{\sin 32°3'}{\cos \sin^{-1} -.11223}, \qquad \theta' = 32°16'$$

and in equation (3.29)
$$\delta_2' = \tan^{-1} \frac{-.11223}{-.99180} = 6°30'$$

and in equation (3.29')
$$\sin \theta'' = \frac{\sin 32°3'}{\cos \sin^{-1} .060293}, \qquad \theta'' = 32°7'$$

so that in equation (3.25)

$$g_1 = 32°16' + 3°28.7' = 35°44.7'$$

Similarly

$$g_2 = 32°7' + 6°30' = 38°37'$$

$$g_3 = 32°16' - 3°28.7' = 28°47.3$$

$$g_4 = 32°7' - 6°30' = 25°37'$$

In the case (d) of an atomic plane which intersects the plate face in a line which is neither parallel nor normal to the plane of the instrument, the angle δ that the atomic normal makes with the plane of the instrument will be different for different positions of the plate.

If N_V is the direction cosine of the normal to the atomic plane with reference to that plate edge (P axis) that is placed in the vertical position,

$$\delta = \cos^{-1}N_V - 90° = -\sin^{-1}N_V$$

This value of δ may be used in determining β according to formula (3.3). For example, when the P_3 axis (width) of an NT plate is placed parallel to the axis of the instrument,

$$\delta = -\sin^{-1}N_3 = -\sin^{-1}.060293 = -3°27.4'$$

(The negative sign indicates deflection of the normal toward the negative end of the P_3 axis and may be disregarded in determination of β).

Whence $\sin \beta = 2 \sin 32°03' \sin 3°27.4'$

$$\beta = 3°40'$$

This means that the beam reflected from the $(2\bar{1}\cdot3)$ atomic plane when the NT plate is placed with its P_3 (width) axis parallel to the axis of the instrument would be received by an ionization chamber which would accept a beam making an angle of $3°40'$ with the plane of the instrument.

When the P_1 axis (length) of an NT plate is placed parallel to the axis of the instrument

$$\delta = \cos^{-1}N_1 - 90° = \sin^{-1} -.11223 = -6°26.7'$$

Since this is a larger β value than most ionization chambers will accept, the $(2\bar{1}\cdot3)$ plane cannot be used in most cases to check an NT plate with its P_1 axis parallel to the axis of the instrument unless the ionization chamber is moved vertically.

(b) and (c) Atomic plane intersecting plate-face in a line which is either normal or parallel to the plane of the instrument.

For plates rotated about X only (as AT, BT, CT and DT) the problem

is so simple (See section 3.6, b) that the above procedure need not be followed.

For such plates the complete transformation is an A_2 rotation around X:

$$r''' = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos A_2 & -\sin A_2 \\ 0 & \sin A_2 & \cos A_2 \end{pmatrix} \tag{3.38}$$

The planes used for checking these plates are of the type $(0k \cdot \ell)$ so that, from equations (3.13) and (3.14)

$$N = \begin{pmatrix} 0 \\ 2k/\sqrt{3} \\ \ell/1.1 \end{pmatrix} \frac{1}{\sqrt{S}} \tag{3.39}$$

whence

$$r'''N = \begin{Bmatrix} 0 \\ \dfrac{2}{\sqrt{3}} k \cos A_2 \dfrac{\ell}{1.1} \sin A_2 \\ \dfrac{2}{\sqrt{3}} k \sin A_2 + \dfrac{\ell}{1.1} \cos A_2 \end{Bmatrix} \frac{1}{\sqrt{S}} \tag{3.40}$$

but $\dfrac{2K}{\sqrt{3}\sqrt{S}} = \sin \gamma_{0k \cdot \ell}$ and $\dfrac{\ell}{1.1\sqrt{S}} = \cos \gamma_{0k \cdot \ell}$

where γ = the angle between the normal to the atomic plane and the Z axis (note that for γ angles on the negative side of the Z axis the value given in Table I should be subtracted from $180°$, as in the case of the AT cut given below).

Thus (3.40) may be written

$$N = \begin{pmatrix} 0 \\ \sin (\gamma - A_2) \\ \cos (\gamma - A_2) \end{pmatrix} \tag{3.41}$$

and (3.25) becomes

$$g_1 = \theta - \delta_1' \quad \text{where} \quad \delta_1' = 90° + A_2 - \gamma \tag{3.42}$$

(Note that where A_2 is positive γ will be on the negative side of the Z axis)

whence

$$g_1 = \theta + \gamma - 90° - A_2 \tag{3.43}$$

$$g_3 = 90° + \theta + A_2 - \gamma \tag{3.44}$$

For position 2, equation (3.40) applied to equation 3.27 gives

$$\sin \theta = \sin (\gamma - A_2) \sin g_2$$

so that we may write

$$\sin g_2 = \sin g_4 = \frac{\sin \theta}{\sin (\gamma - A_2)} \qquad (3.45)$$

As an example consider a BT plate for which $A_2 = -49°$, corrected around the WW axis (Fig. 3.12) from the $(02 \cdot 3)$ plane. Table I gives us $\theta_{023} = 34°05'$, $\gamma_{023} = 40°16'$ whence

$$g_1 = 34°05' + 40°16' - 90° + 49° = 33°21'$$

$$g_3 = 90° + 34°05' - 49° - 40°16' = 34°49'$$

$$g_2 = g_4 = \sin^{-1} \frac{\sin 34°05'}{\sin 89°16'} = 34°05'$$

For an AT plate of $A_2 = +35°15'$ corrected around the WW axis from $(01 \cdot \bar{1})$ we find, under $(01 \cdot 1)$, that $\theta = 13°20'$, $\gamma = 180 - 51°47'$ whence

$$g_1 = 13°21' + 180° - 51°47' - 90° - 35°15' = 16°18'$$

$$g_3 = 2\theta - g_1 = 10°22'$$

$$g_2 = g_4 = \sin^{-1} \frac{\sin 13°20'}{\sin 92°58'} = 13°21'$$

A complete determination of the errors in a quartz plate includes the measurement of angle errors about *three* mutually perpendicular axes such as, for example, the plate edges. Therefore, in correcting a plate we must use three different X-ray "shots"; usually two on the major surface with the plate rotated 90° between the two "shots" and a third on a surface normal to the major surface, commonly called an "edge". If the plate is thin, the intensity of reflection from the "edge" is low and this measurement becomes difficult.

The goniometric procedure described above is best adapted to the routine measurement of errors in large numbers of plates which are known to be close to the correct orientation. If the error in a plate is large, however, or if the cut is a rare one or the orientation of the plate completely unknown, analysis by the goniometric method may be extremely difficult.

A simple, direct method of determining the orientation of such plates is that of the Laue camera.

3.10 Use of Laue Photographs in Determining the Orientation of a Quartz Plate

In the original Laue photograph the X-ray beam passed through the crystal and was diffracted so as to give a spot pattern on a photographic

plate beyond the crystal. This necessitated either the use of a very thin crystal or a long exposure.

To avoid the variation due to crystal thickness we adopted the "Back Reflection Laue camera." As shown in Fig. 3.23 the X-ray beam passes through a hole in the photographic film before striking the crystal. It is collimated by two pinholes, one on each side of the film. Spot reflections from many planes fall on the film and in a few minutes exposure leave a record of their points of impingement. Most of these reflections are not due to the peaks of the radiation curve (Fig. 3.2) but each spot is due to a
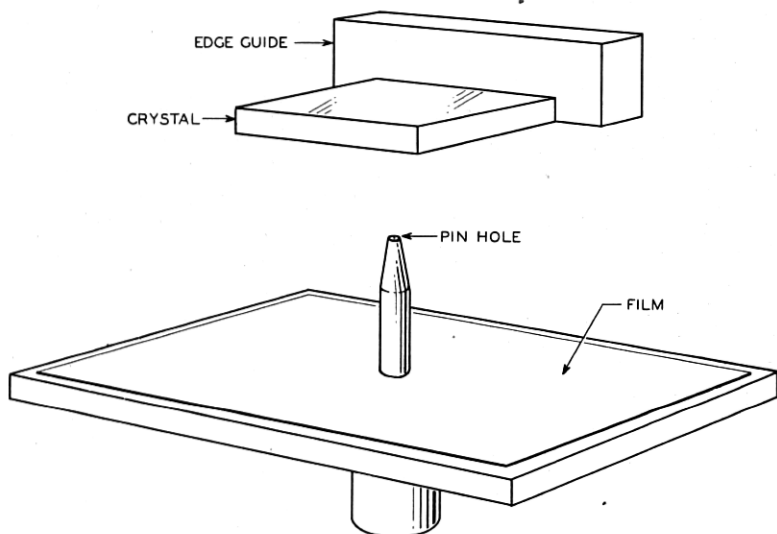

Fig. 3.23—Arrangement of collimator, film and crystal plate for a back reflection Laue photograph

different λ from the continuous background of Fig. 3.2. However, each satisfies the equation $n\lambda = 2d \sin \theta$.

Figure 3.24 is such a record with many of its spots marked. The spot $(01 \cdot 1)$ is recognized as the point of intersection of the greatest number of rows of spots. The spot $(01 \cdot 2)$ is the second most obvious intersection point.

On examining such a film we recognize such spot configurations and then mark the indices of the corresponding atomic plane for a few chosen spots. From these we can measure three angle-errors. For example, if the crystal, Fig. 3.25, is rotated about the vector t by amount e_t the spot pattern on the film will be rotated by the same amount $e_{t'}$. If the crystal is in error by amount e_w being rotated about ww clockwise the spot pattern of the film

will be shifted to the left and the value of $e_{w'}$ can be measured by means of a specially graduated scale. See Fig. 3.24. (Scale used is of Lucite and
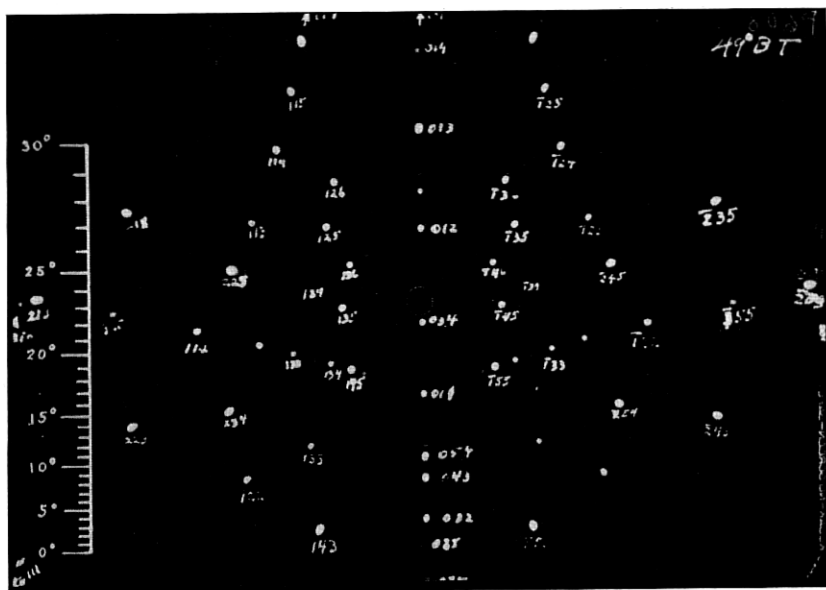

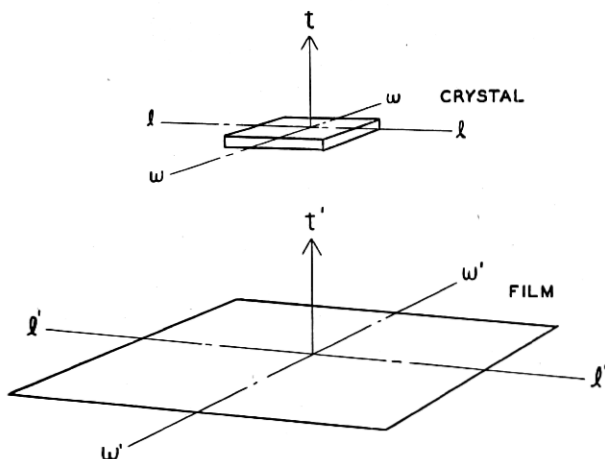Fig. 3.24—Back reflection Laue photograph of a BT plate

Fig. 3.25—Relation of displacement directions on the film to the ℓ–ℓ and w–w axes

calibrated to 10 minutes.) Similarly, if the crystal is misoriented about $\ell\ell$ by amount e_ℓ the spot pattern will shift along $w'w'$ and $e_{\ell'}$ can be measured by the same scale used on $e_{w'}$. To compensate for film expansion due to

changes in temperature and humidity, measurements are made from two spots about equally removed from the center but on opposite sides.

If all three errors exist simultaneously they may still be measured in this way provided they are small.

Given e_ℓ, e_w and e_t for plates containing the X axis the errors in A_1, A_2 and A_3 can be computed by the formulae

$$\text{Error in } A_1 = e_w/\cos A_2$$

$$\text{Error in } A_2 = e_\ell$$

$$\text{Error in } A_3 = e_t - e_w \tan A_2$$

Consider, for example, the negative (Fig. 3.24) for a *BT* plate. The row of spots $(01\cdot4)$, $(01\cdot3)$, $(01\cdot2)$, $(03\cdot4)$, $(01\cdot1)$, etc., results from reflections from the group of planes parallel to the X axis (see Fig. 3.8). (Such a group of planes all parallel to a given line is called a zone.) In the *BT* cut the A_2 angle results from rotation around the X axis. The effect of such a rotation on the photograph (Fig. 3.24) is a shift of the spots of the $(0k\cdot\ell)$ atomic planes along the line on which they lie. Therefore, to measure the A_2 angle from Fig. 3.24, we choose two spots along this line about equidistant from the center of the film such as $(01\cdot1)$ and $(01\cdot2)$ and measure the angular distance α between the incident X-ray beam and the reflected ray causing each spot by measuring the distance between the center of the film and each spot using the specially calibrated scale.

By "center of the film" is meant the point through which the center of the X-ray beam passed. This point is located as follows. When the film is in place in the camera, two reference points are pricked in it by pins built into the camera. These points may be seen in Fig. 3.24 as small white circles near the center of the right and left edges of the picture. The center of the X-ray beam is at the center of a line between these two points.

When the negative is to be measured it is placed on a glass plate engraved as shown in Fig. 3.26 with the reference points at P, P. The intersection X then marks the center of the film.

If A_2 is the plate angle around the X axis from the Z axis and γ is the angle between the atomic plane normal and the Z axis and α is the angle measured between the center of the film and the spot, then

$$A_2 = 90° - \gamma_{hk\cdot\ell} + \alpha_{hk\cdot\ell} \tag{3.46}$$

For the two spots chosen for measurement of the *BT* plate of Fig. 3.24,

$A_2 = 90° - \gamma_{01\cdot2} - \alpha_{01\cdot2}$ (Rotation from the *BT* cut to the $01\cdot2$ plane is a negative rotation)

$A_2 = 90° - \gamma_{01\cdot1} + \alpha_{01\cdot1}$

$$A_2 = 90° - \frac{\gamma_{01\cdot2} + \gamma_{01\cdot1}}{2} + \frac{\alpha_{01\cdot1} - \alpha_{01\cdot2}}{2}$$

and substituting the values of $\gamma_{01\cdot2}$ and $\gamma_{01\cdot1}$ from Table I,

$$A_2 = 90° - \frac{32°25' + 51°47'}{2} + \frac{\alpha_{01\cdot1} - \alpha_{01\cdot2}}{2}$$

$$= 47°54' + \frac{\alpha_{01\cdot1} - \alpha_{01\cdot2}}{2}$$

(3.47)

For more complicated cuts, such as the NT cut, four spots are chosen, two close to the line $\ell'\ell'$ and two close to the line $w'w'$. (See Fig. 3.25). The positions these spots should occupy on the film for a correct NT cut
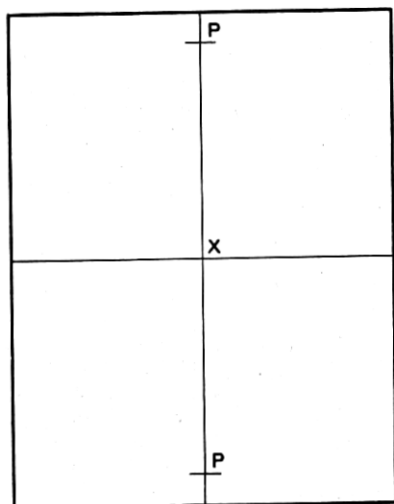

Fig. 3.26—Engraved glass plate for measuring Laue photographs

are computed and compared to the spot-positions on the measured film. From this comparison the following errors are determined:

e_w error in rotation around the ww axis of the plate from the average displacement along $\ell'\ell'$.

e_ℓ error in rotation around the $\ell\ell$ axis of the plate from the average displacement along $w'w'$.

e_t error in rotation around the t axis by the average angular displacement of the four spots about the center of the film.

The first step in determining the position of a spot for any given atomic plane in a correctly cut plate is the determination of the direction cosines of the normal to that atomic plane with respect to the plate edges. This procedure has been described in Section 3.8.

Figure 3.27 is a Laue photograph of an NT plate. Taking this as an example, we may choose the four spots $(31\cdot4)$, $(3\bar{1}\cdot5)$, $(2\bar{2}\cdot3)$ and $(5\bar{2}\cdot5)$

from which to make the measurements. From equations (3.13) and (3.9) we find that the direction cosines of the normals to these atomic planes in terms of the NT plate edges are

$$(31 \cdot 4) = \begin{pmatrix} .42561 \\ -.90357 \\ .04972 \end{pmatrix}_P, \quad (3\bar{1} \cdot 5) = \begin{pmatrix} -.01112 \\ -.99294 \\ -.11812 \end{pmatrix}_P,$$

$$(2\bar{2} \cdot 3) = \begin{pmatrix} -.42618 \\ -.90421 \\ .02816 \end{pmatrix}_P, \quad \text{and} \quad (5\bar{2} \cdot 5) = \begin{pmatrix} -.00898 \\ -.99140 \\ -.13062 \end{pmatrix}_P$$
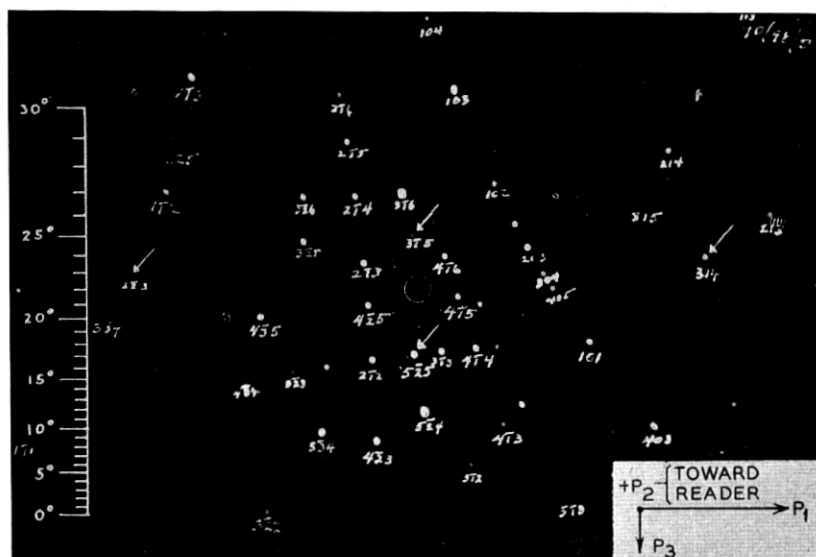

Fig. 3.27—Back reflection Laue photograph of an NT plate

The orientation of the unit normal to the atomic plane $(31 \cdot 4)$ with respect to the plate edges is shown in Fig. 3.28. Since $.90357 = \cos 25°22'$, the angle between the unit normal N and the P_2 axis is $25°22'$. Since $\dfrac{.04972}{.42561} = \tan 6°40'$ the angular component of the unit normal N in the P_1P_3 plane is $6°40'$ that is, the $(31 \cdot 4)$ spot should lie in a direction $6°40'$ from the long axis of the film and should be at a distance from the center corresponding to $25°22'$ as read from the special scale.

The correct locations of the other three spots are similarly calculated. These values are given below, with the values actually measured from the Laue photograph of the NT plate and the errors determined from a comparison of the calculated and measured values.

(n.b. clockwise and counter-clockwise directions are as seen when looking toward the − end of the axis of rotation)

Distance from the Center

	Calculated	Measured	Difference	Error
(31·4)	25°22′	25°22′	0′	0′
(2$\bar{2}$·3)	25°17′	25°18′	+1′	
(3$\bar{1}$·5)	6°49′	6°18′	−31′	30′ counter-clock-wise about P_1
(5$\bar{2}$·5)	7°31′	8°00′	+29′	

Azimuth
(counter-clockwise from $+P_1$)

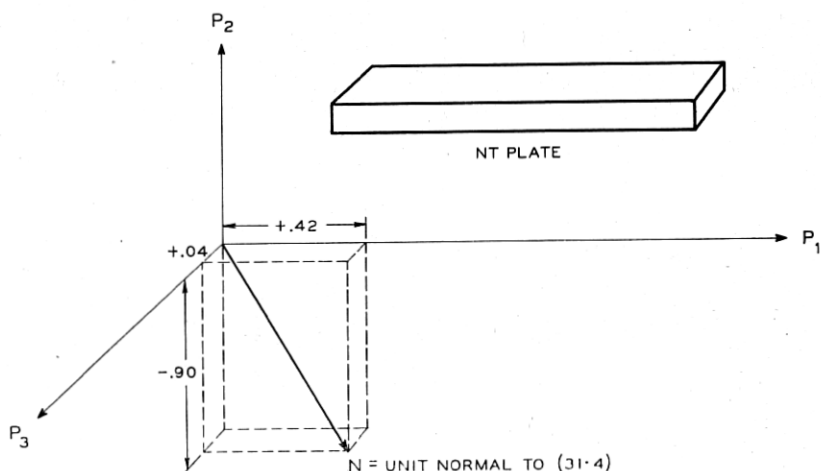|	Calculated	Measured	Difference	Error
(31·4)	6°40′	5°48′	−52′	7′ counter-clock-
(2$\bar{2}$·3)	176°12′	177°18′	+66′	wise about $-P_2$
(3$\bar{1}$·5)	95°25′	95°36′	+11′	or clockwise about
(5$\bar{2}$·5)	266°03′	267°06′	+63′	$+P_2$ [8]

Fig 3.28—Orientation of the (31.4) atomic plane in the NT plate

Now, a small rotation of amount E_1 counter-clockwise about P_1 followed by one of amount E_2 counter-clockwise about P_2 and a similar one E_3 about P_3 is given to the first order approximation by the matrix:

$$e = \begin{pmatrix} 1 & -e_3 & e_2 \\ e_3 & 1 & -e_1 \\ -e_2 & e_1 & 1 \end{pmatrix} \tag{3.48}$$

Where e_1, e_2 and e_3 are the sines of the rotation angles E_1, E_2, E_3.

[8] This error is determined by averaging the "difference" values for the two planes.

From (3.48) we see that the order of application of e_1, e_2, e_3 is immaterial.

If the desired transformation is $r°$ (i.e., with zero errors) ($=r'''$, equation 3.9 for the NT plate), and the actual one is r, we can consider that $r°$ is made up from the desired rotations $A_1°$, $A_2°$, $A_3°$ and the r is made up of the rotations $A_1 = A_1° + \Delta_1$, $A_2 = A_2° + \Delta_2$, $A_3 = A_3° + \Delta_3$; or alternatively r is made from the transformation $r°$ followed by the transformation e so that

$$r = er° \tag{3.49}$$

whence

$$-\tan A_1 = \frac{(er°)_{21}}{(er°)_{22}} = \frac{r_{21}° - e_1 r_{31}° + e_3 r_{11}°}{r_{22}° - e_1 r_{32}° + e_3 r_{21}°}$$

$$-\sin A_2 = (er°)_{23} = r_{23}° - e_1 r_{33} + e_3 r_{13}° \tag{3.50}$$

$$-\tan A_3 = \frac{(er°)_{13}}{(er°)_{33}} = \frac{r_{13}° - e_3 r_{23}° + e_2 r_{33}°}{r_{33}° - e_2 r_{13}° + e_1 r_{23}°}$$

where $(er°)_{21}$ is the term in row 2, column 1 of the matrix $er°$; $r_{23}°$ is the term in row 2, column 3 of the matrix $r°$ (equation 3.9), etc.

From our unfinished example of the NT plate we have $e_1 = .0087$, $e_2 = -.0018$, $e_3 = 0$ whence

$$-\tan A_1 = \frac{-.64279 + .0087 \times .766 + 0}{-.10662 - .0087 \times .08946 + 0} = \frac{-.6361}{.1074}$$

whence $A_1 = 99°36'$

$$-\sin A_2 = -.75852 - .0087 \times .6365 + 0 = -.7640$$

whence $A_2 = 49°49'$

$$\tan A_3 = \frac{-.13917 - 0 - .0018 \times .6365}{.63653 + .0018 \times .1392 - .0087 \times .7585} = \frac{-.1403}{.6302}$$

whence $A_3 = -12°33'$

In starting work on a new cut of crystal one may have difficulty in finding the indices of the Laue spots. The easiest method is to photograph a crystal that has been carefully cut at measured angles from known planes (for instance natural faces). For example, from the angles laid off in the shop an NT plate such as that described should be sufficiently accurate that when P_2 is located on the atomic plane chart, Fig. 3.7, and several nearby planes of small indices are computed on the P axes, there should be no doubt as to which spots correspond to these locations.

From a few of these spots one can find many others by "zonal" relations. A zone is a family of atomic planes all of which are parallel to one line called the zonal axis. Just as there are indices of a plane there are zonal indices.

Two planes determine a zone. The zonal indices are computed by "cross multiplication" of the indices of the planes. The two planes $(h_1k_1 \cdot \ell_1)$ and $(h_2k_2 \cdot \ell_2)$ determine the zone $(k_1\ell_2 - \ell_1k_2,\ \ell_1h_2 - h_1\ell_2,\ h_1k_2 - k_1h_2)$. In practice this is developed by writing the indices of each plane twice; those of one under those of the other, then striking out the two end members of each and taking the difference of the cross products. Whereas plane indices are always enclosed in parentheses ( ), zonal indices are always enclosed in brackets [ ]. For example $(0\ 0\cdot1)$ and $(0\ 1\cdot0)$ are in the zone:

$$
\begin{array}{c|cccc|c}
0 & 1 & 0 & 0 & 1 & 0 \\
 & & \times & \times & \times & \\
0 & 0 & 1 & 0 & 0 & 1 \\
\hline
 & & \multicolumn{2}{c}{[1\ 0\cdot0]} & &
\end{array}
$$

while $(2\bar{1}\cdot0)$ and $(21\cdot2)$ are in the zone $[24\cdot\bar{4}]$, or reducing, in the $[12\cdot\bar{2}]$ zone.

This zonal relation is reciprocal in the sense that the plane common to two zones is derived in the same way as were the zonal indices. For example, the plane common to the zones $[10\cdot0]$ and $[12\cdot\bar{2}]$ is

$$
\begin{array}{c|cccc|c}
1 & 2 & \bar{2} & 1 & 2 & \bar{2} \\
 & & \times & \times & \times & \\
1 & 0 & 0 & 1 & 0 & 0 \\
\hline
 & & \multicolumn{2}{c}{(0\ \bar{2}\cdot\bar{2})} & &
\end{array}
$$

or, $(0\ \bar{1}\cdot\bar{1})$

If a given face with indices xyz lies in a zone with the symbol uvw, the following equation, known as "the zonal equation" will be true:

$$ux + vy + wz = 0$$

In the Laue photograph of the BT plate, Fig. 3.24, the row of spots $(12\cdot2)$ $(13\cdot3)$ $(01\cdot1)$ $(\bar{1}5\cdot5)$ $(\bar{1}3\cdot3)$ etc. is due to reflections from planes which belong in one zone. Other rows such as $(\bar{1}2\cdot5)$ $(\bar{1}2\cdot4)$ $(\bar{1}2\cdot3)$ etc. cross this row at some spot whose indices can be computed if two spots in each row are known. If the row passes through the center of the film all the spots lie on a straight line. If the row is off center it is curved, convex towards the center. Experience soon shows us how much curvature goes with how great a distance from the center for a series of spots to form a zone.

3.11 X-Ray Checks of Slabs in the Course of Manufacturing Quartz Plate

In the course of manufacturing a quartz plate it is common practice to X-ray check the first sawn slab and correct the orientation of the quartz with respect to the saw before cutting the remaining slabs. Each of these slabs is then cemented to the head of a barrel jig so built that the orientation

of the head can be adjusted through small angles and clamped in the adjusted position. The jig is then placed in an X-ray goniometer set for reflection from the plane to be used and the slab adjusted until maximum reflection is obtained. The jig itself is not moved. If the slab were perfectly cut the maximum reflection would be obtained when the head of the jig was normal to the jig axis. A miscut will give maximum reflection at some other angle and, when clamped at this angle of maximum reflection,
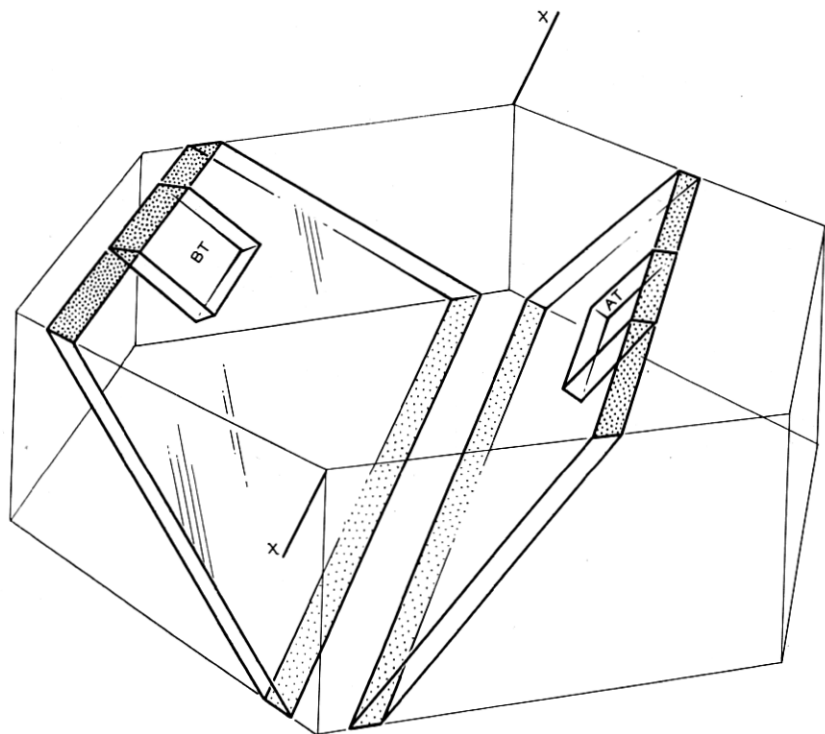

Fig. 3.29—Orientation of AT and BT plates and slabs with respect to Z section ($+X$ toward reader for right-handed quartz)

will have a surface ground on it normal to the axis of the jig. This surface will have the desired orientation of the plate.

As in the case of the finished plate the slab must be checked for angle-error around two mutually perpendicular axes in the plate surface. The intersection of the slab surface with the Z-cut surface is commonly taken as one axis and the normal to this as the other.

For the AT and BT cuts these two axes are the same ones used in correcting the finished plate and the same settings for jig holder and ionization chamber may be used (See Fig. 3.29). For any cut with an A_3 angle that

is not 0°, such as the *NT* cut, these two axes will not be the same as those used in checking the finished plate and different settings of the jig-holder and ionization chamber will have to be calculated. The procedure is similar to that for the determination of the *g* angles for the finished plate except that the components of the unit vector of the atomic plane used are multiplied by the matrix which expresses the transformation after two rotations (A_1 and A_2) (equation 3.7) instead of the matrix for three rotations.
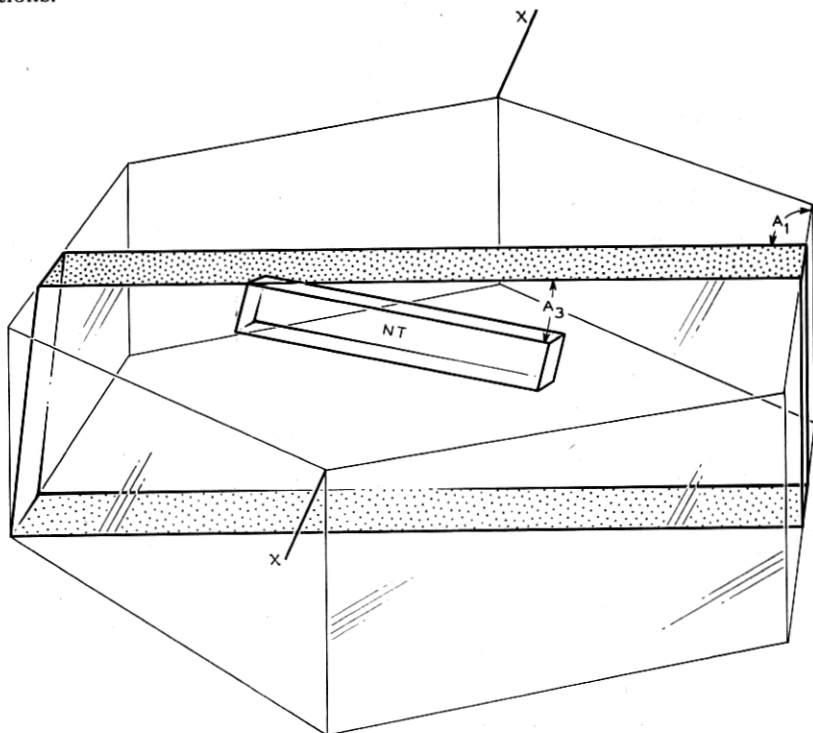

Fig. 3.30—Orientation of NT plate and slab with respect to Z section (+X toward reader for right-handed quartz)

For example, in the case of the *NT* cut, $A_1 = 99°25'$ and $A_2 = 49°20'$ so that the $A_1 A_2$ matrix (equation 3.7) becomes

$$r'' = \begin{pmatrix} -.1636 & .9865 & 0 \\ -.6429 & -.1066 & -.7595 \\ -.7483 & -.1241 & .6517 \end{pmatrix}$$

which, multiplied by the components of the unit normal to the atomic plane

($2\bar{1}\cdot3$) in terms of the X, Y, Z axes $\begin{pmatrix} .5914 \\ 0 \\ .8064 \end{pmatrix}$ gives the components of the unit

normal to the atomic plane $(2\bar{1}\cdot3)$ in terms of the reference edges of the slab after rotations A_1 and A_2 (See Fig. 3.30).   These are

$$N = \begin{pmatrix} -.0966 \\ -.9919 \\ -.0830 \end{pmatrix}$$

Substituting in equation (3.26)

$$\tan \delta_1' = \frac{-0830}{.9919}$$

$$\delta_1' = -4°48'$$

and in equation (3.29)

$$\tan \delta_2' = \frac{-.0966}{-.9919}$$

$$\delta_2' = 5°35'$$

and from equations (3.25), (3.28), (3.31), and (3.35):

$$g_1 = 27°25'$$

$$g_2 = 37°45'$$

$$g_3 = 37°1'$$

$$g_4 = 26°35'$$

which are the g angles for X-raying the NT slab where the reference edge is the intersection between the slab-surface and the Z-section surface.