PRECISE ANGULAR CONTROL OF QUARTZ-CUTTING BY X-RAYS*

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ABSTRACT

Methods for precise control of the cutting angles of quartz by *x*-ray measurements are described. The quartz must first be oriented by some other method (usually optically) before the *x*-ray method can be applied. The sense of direction of the cut is indicated by an arrow drawn on the outer surface of the test cut at the saw and this direction is preserved in making the *x*-ray measurement. The *x*-ray technique is an adaptation of the Bragg ionization chamber method and involves measuring the angle between the surface of the test cut or blank and an atomic reference plane parallel (or nearly parallel) to the surface. All measurements are direct, and require no computation. A Geiger-Muller tube operated in the proportional counter region is employed. Accuracy of the method is approximately $\pm 1.5'$, the measurement requires about 10–15 seconds, and is used by unskilled help. The procedures for calibrating the *x*-ray goniometer and measuring various types of cuts are described in detail. Methods involving precise angular adjustments of sections approxi-

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mately oriented by other methods and the use of reflection intensity differences of certain planes on either side of the optic axis for detecting usable portion of electrical twins and negative and positive directions from Z are described. The methods are applicable to other fields.

Introduction

The mass production manufacture of quartz oscillator-plates requires control of angles of cutting within quite narrow limits. The tolerances generally required for AT- and BT-cuts are $ZZ'\pm 10'$ and $XX'\pm 15'$ from the absolute values. The optical and etch techniques generally used in the preliminary orientation of quartz do not have this required precision. The *x*-ray techniques described below have proven to be the only economical and precise method known today for this purpose. This precision is required in modern oscillator-plates which are manufactured to meet high performance standards. Such properties as the temperature coefficient, frequency-thickness constant, coupling between various modes of vibration, etc., are critically dependent upon orientation and hence the general rule is not to depart greatly from the specified angles, in order to produce blanks with uniform properties.

Before the introduction of *x*-rays, manufacturers oriented by morphology or optical means and cut a test piece from the crystal. This test piece was fashioned into an oscillator and the frequency change with temperature determined. The correction to be applied to the saw was determined from these data but was generally inaccurate because of broadness of the turning point of the temperature-frequency curve, compound angular errors, and other factors. In any case, the test operation usually required about an hour during which time the saw remained idle. No real progress was made because there was no way to correlate precisely the physical porperties with orientation.

This state of affairs may be contrasted with that prevailing today when *x*-rays are used to make hundreds of thousands of measurements accurate to a few minutes every day by untrained help, taking 10 to 15 seconds for each measurement! In fact, this marks the first application of *x*-ray diffraction as an integral part of mass production manufacturing. The natural crystal faces, optical properties and light figures of etched sections serve for preliminary orientation to within a few degrees, but *x*-rays are used to assure precision in cutting to minutes. Indeed without *x*-rays, the mass production manufacture of quartz oscillator-plates with the present extraordinary standards of performance would be impossible.

The great usefulness of *x*-rays lies in the ability to use atomic planes within the quartz to bring a crystal to precise orientation when its natural faces have been sawn away. In actual practice a test sliver is cut on the saw and the angle between the surface and a reference atomic plane such as $(01\bar{1}1)$ for AT-cuts or $(20\bar{2}3)$ for BT-cuts is measured directly and the angular adjustments are then made at the saw. In all cases, however, preliminary orientation by some means must be provided to identify or set up the atomic planes close to the reflecting position.[1] The method also requires coordination between the saws and x-ray settings to preserve the sense of direction.

Acknowledgments. The Geiger-Muller counter tube used for x-ray measurements and its associated electronic circuit were developed by Dr. Herbert Friedman, Naval Research Laboratory, Anacostia Station, Washington, D. C. Without this timely development, the x-ray technique would not have become the invaluable tool for the manufacture of quartz oscillator-crystals. Dr. Friedman also prepared the x-ray powder spectrum of quartz shown in Fig. 11. The x-ray goniometer is based upon a design developed in collaboration with Bendix Radio Corp., Crystal Division, Baltimore, Md. The photographs were taken by Mr. John Derbyshire, N.A.P.

X-RAY APPARATUS

The x-ray apparatus in use by the quartz oscillator industry is an adaptation of the original Bragg ionization spectrometer. The great advantage of the instrument is its precision, as well as its simplicity of operation. Two commercial machines have been widely used (some 300 x-ray machines are in use in the crystal industry). The General Electric Company's apparatus employs a water cooled x-ray tube, drawing comparatively high current and uses an ionization chamber for detection. The North American Philips Co., Inc., manufactures a special unit, described below, (Figs. 1–4), for quartz crystal work in which a low current (3–4 ma.) air cooled x-ray tube is used with a high voltage, high sensitivity Geiger counter tube.[2] Both machines have two-window tubes and are equipped with an x-ray goniometer for each window.

Several types of x-ray crystal holders are available (Figs. 2, 5–8) for

[1] Parrish, William and Gordon, Samuel G., Orientation techniques for the manufacture of quartz oscillator-crystals: *Am. Mineral.*, this issue; Cutting schemes for quartz crystals: *ibid.*

[2] The normal precautions in the use of x-ray apparatus should be followed as prescribed in National Bureau of Standards Handbook HB20, *X*-Ray protection, July 24, 1936, Supt. of Documents, Washington, D. C. The commercial apparatus is shock- and ray-proof. The on-off shutter must be closed before placing or removing the crystal from the holder. In some crystal plants, orientation of crystals mounted on jigs is attempted by x-rays. In several instances the operators get their fingers in the x-ray beam while making adjustments on the jigs. The carrying of dental films by the operator, while useful, does not indicate that the fingers are not getting in the beam.

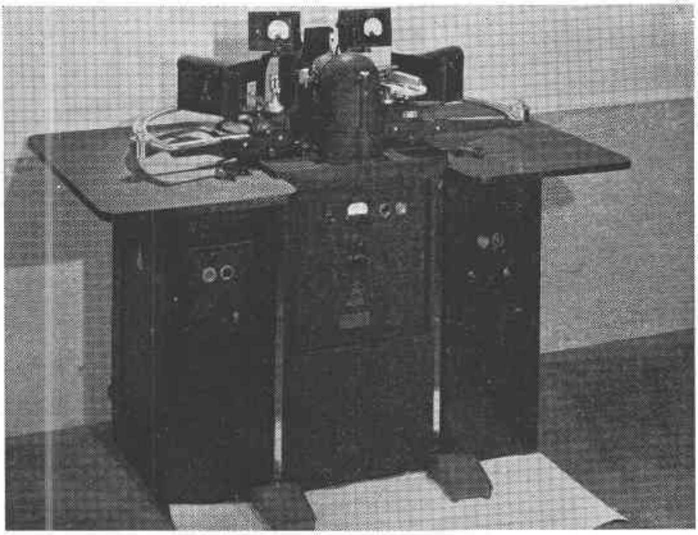


Fig. 1. North American Philips Co., Inc. *X*-ray machine for quartz crystals. Power supplies for each Geiger-Muller tube are in cabinets on either side of high voltage transformer and controls are contained in middle section.

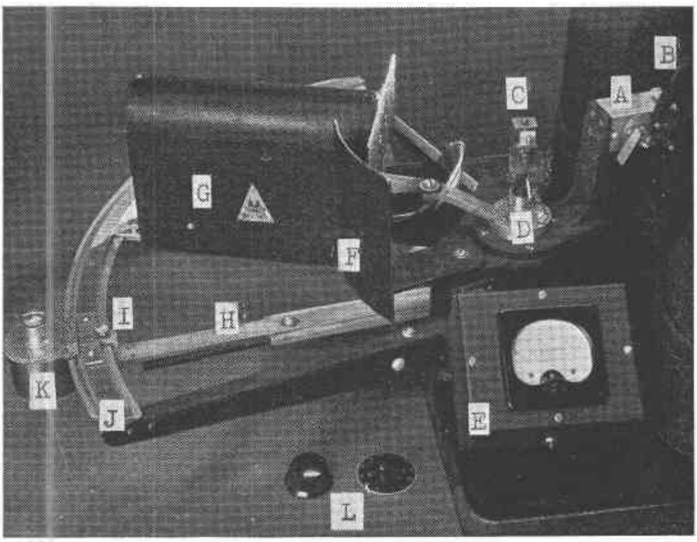


Fig. 2. *X*-ray goniometer. (A) Slit system, (B) on-off shutter, (C) crystal holder for measuring blanks, (D) collar for tightening crystal holder to goniometer arm, (E) 0–1 milliammeter for indicating *x*-ray reflections, (F) direct beam shield, (G) Geiger-Muller tube housing, (H) goniometer arm, (I) auxiliary scale, (J) scale graduated in 1° steps, (K) minute scale, (L) step and microswitch for changing voltage on G.-M.

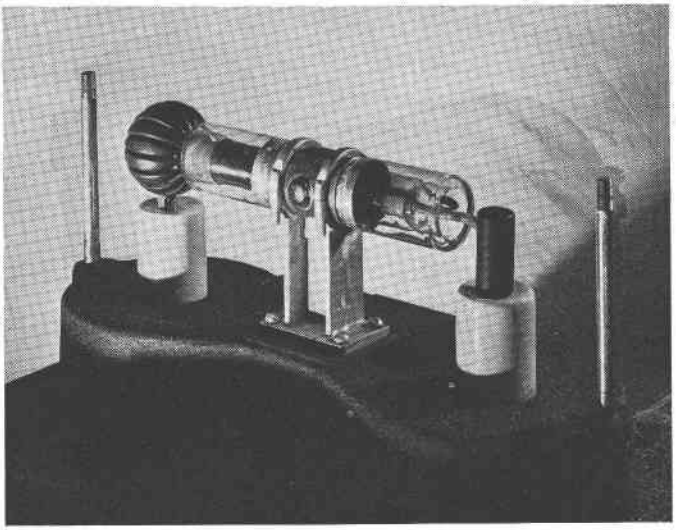


FIG. 3. Air-cooled x-ray tube with copper target and two Lindemann glass windows. Shadow shows position of protective housing.

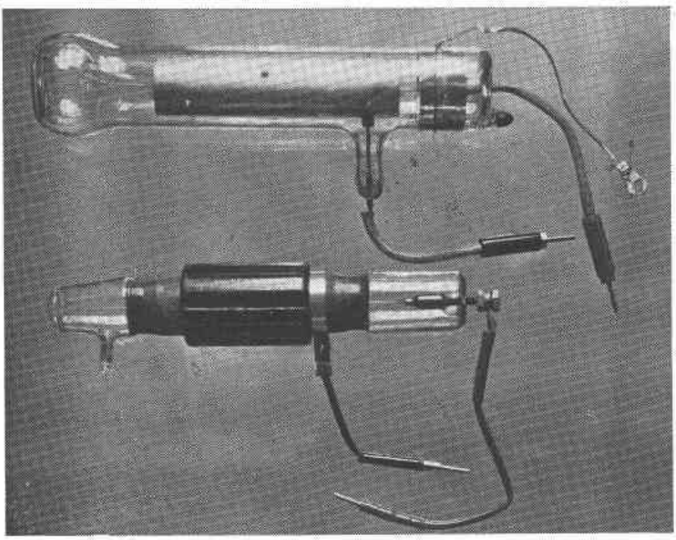
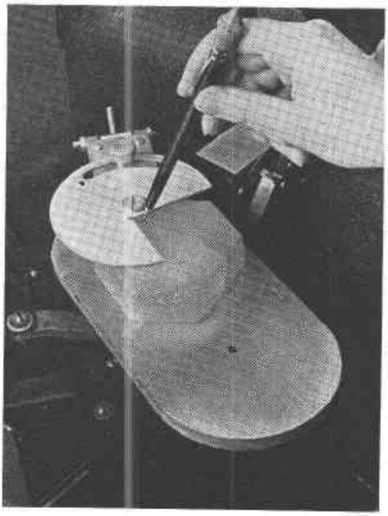


FIG. 4. Geiger-Muller tubes. Older type with thin bubble window above; newer type with Lindemann glass window and metal casing below.



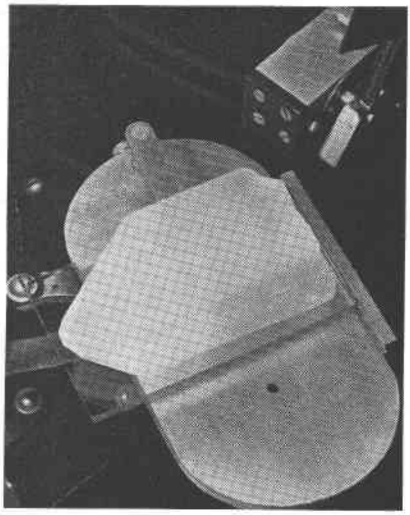
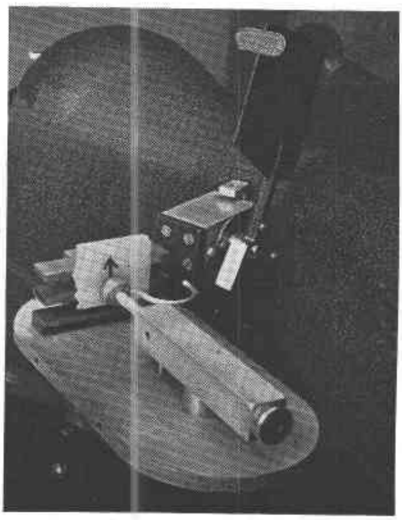


FIG. 5. *X*-ray table and marking guide for locating X- or Y-axes in Z-sections.
FIG. 6. *X*-ray table for measuring deviations of mounted crystal or X-block with respect to reference edge of glass mounting plate.



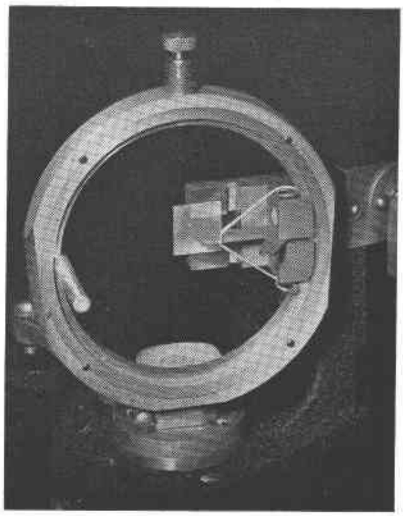


FIG. 7. *X*-ray table for measuring angular deviations of test cuts. Plunger arrangement allows rapid setting of quartz with irregular back.
FIG. 8. Rotating blank holder sometimes used for measuring ZZ′ and XX′ by rotating blank 90° after first measurement.

the various operations. In all, the test cut piece or blank is placed flush against the reference surface of the crystal holder of the x-ray goniometer. The crystal is held in place by a clip and spring. It is apparent that the anvil of the crystal holder must be perfectly flat to obtain accurate measurements. Although hardened tool steel, and sometimes boron carbide are used, the wear on the surface produced by constant use requires careful checking of the surface every few days. The crystal holder is locked to a ruled arm which is marked in degrees and a circular scale which reads directly to minutes. The Geiger tube setting is fixed and the holder with crystal rotated in the x-ray beam until maximum deflection of a milliammeter indicates the chosen atomic planes are in the position of maximum reflection. The setting is then read directly from the ruled arm and minute scale.

Geiger-Muller Counter Tube for X-Rays. It will be apparent from the description of the mass production techniques for manufacturing quartz crystals that film methods would have been totally inadequate. One can readily understand how production could not have reached its peak if the saw remained idle while a film of the test cut was being exposed, developed and measured. Fluorescent screen methods require a darkened room and have been used by few plants. Methods involving the use of pairs of reflection spots on fluorescent screens are confined only to the setting of quartz on a jig. Techniques for obtaining precise angular measurements by this means are not in use. Various ionization chamber techniques with sensitive galvanometers were tried but were generally not practical in the industry because of the small currents involved, vibration and humidity problems, critical adjustments, etc. The most successful device developed is the Geiger-Muller counter and associated electronic circuits developed at Naval Research Laboratory.[3]

The G.-M. tube, Fig. 4, is designed for high efficiency for the characteristic x-ray wave-length being used. It is filled with argon at nearly atmospheric pressure and a small amount of ethyl alcohol to make the tube self-quenching. This fast counter has extraordinarily high sensitivity—far greater than could be obtained with ionization chambers or film. The x-rays reflected from the crystal pass through a thin Lindemann glass window on the G.-M. tube and then through the gaseous area. About 85% of the Cu$K\alpha$ x-rays passing through the window are absorbed in the 10 cm. gas path. Each x-ray quantum produces a number of

[3] Friedman, Herbert; Kaiser, Herman F. and Christenson, Arthur L., Applications of Geiger-Muller counters to inspection with x-rays and gamma rays: *Jour. Am. Soc. Naval Eng.*, **54**, 177–209 (1942). For a general discussion of G.-M. tubes see Strong, John, *Procedures in Experimental Physics*, Prentice-Hall, Inc., New York (1938), Chapter VII, by H. V. Neher.

charged particles which cascade to form many others. These are collected at the anode of the G.-M. tube and recorded as one pulse. The chrome-iron cathode cylinder is sealed directly to the soft glass ends and the anode is a tungsten wire mounted in glass at the end of the tube. The wire is about 0.030″ diameter which permits it to remain suspended in the center of almost the entire length of the tube.

The sensitivity of the G.-M. tube is dependent upon the potential difference applied to the electrodes, since the tubes for quartz work are operated in the proportional counter region; i.e., just below the G.-M. tube plateau. Approximately 800 to 1200 volts is supplied by a constant voltage electronic D.C. power supply housed under the goniometer table;[4] a step switch with micro adjustments allows a rapid adjustment of the voltage. The exact voltage required will vary with the amount of gas in the tube, the reflecting power of the atomic plane, width of slits, slit to specimen to G.-M. distance, etc. In practice it is not necessary to adjust the voltage when making successive measurements on the same type of cut. The G.-M. tube is followed by a simple one-stage amplifier and a 3″ 0–1 milliammeter is used for detecting the current produced. The sensitivity of the apparatus is sufficient to detect all the commonly used atomic reference planes in quartz.

A series of experiments on BT-blanks showed that the x-ray measurements are reproducible to approximately $\pm 1.5'$. Greater accuracy may be obtained using the $(10\bar{1}1)$ atomic planes which give a sharper reflection than $(20\bar{2}3)$ and plotting a curve such as the one shown in Fig. 9.

Alignment Procedure. A piece of 0.0004″ thick nickel foil is attached to the window of the x-ray tube housing to absorb $CuK\beta$. Two fixed slits each with an opening 1/32″ wide, 5/16″ high and 2″ apart and an on-off shutter are contained in a housing which is attached directly to the x-ray goniometer. The position of each slit may be adjusted by two screws each working against one of the edges of the piece of metal containing the slit. These are adjusted and aligned with the window on the x-ray tube housing by shifting the goniometer assembly. The G.-M. tube may be used in this alignment by removing the crystal holder from the path of the beam and placing a few pieces of plate glass in front of the window to reduce the intensity of the direct beam. The x-ray goniometer is then bolted to the x-ray machine table and the direct beam should then be at or very close to 0° on the scale. However, accuracy of this 0° setting is not important for the type of measurements generally used in quartz crystal work.

The crystal holder may be rotated independently of the goniometer

[4] Batteries have also been used. Since very little current is drawn, they practically have shelf life.

arm by loosening the collar which locks it to the goniometer arm. The goniometer arm is set to a position on the scale which is convenient for reading (usually 0°). With the G.-M. housing set at the approximate 2θ for the desired atomic planes and the x-ray standard crystal in place, the holder is rotated by hand to a position of maximum reflection as indicated by maximum deflection on the milliammeter. The collar is then locked so that the goniometer arm can rotate the crystal holder and is accurately adjusted to maximum deflection. Then the G.-M. housing is moved until the milliammeter shows maximum deflection. The position of the G.-M. housing is not critical due to the wide scatter slit and the wide window on the G.-M. tube. The housing may be moved about $\frac{1}{4}°$ before the milliammeter will show a change from maximum deflection. The goniometer arm is then turned to maximum reflection and an auxiliary scale slipped on the goniometer scale and tightened in the new 0° position. The minute scale is set to 0′ by loosening the screw on top of the disc and rotating the scale by hand while holding the goniometer arm in place. The goniometer is now set up to give direct readings with respect to that of the standard crystal used in the alignment. If it is desired to make direct readings from a point on the scale other than the one given by the standard crystal, the goniometer arm is then rotated to the desired angle and the minute scale readjusted to 0′ for this new position. For example, if a $(10\bar{1}1)$ x-ray standard was used for the alignment and it was desired to measure directly the ZZ′ angle of AT-cuts, the goniometer arm would be rotated $38°13' - 35°15' = 2°58'$ clockwise, the auxiliary scale moved to the new position and the minute scale reset to 0′.

The crystal holder and G.-M. tube should be carefully checked for alignment in the horizontal plane with the slit by using a straight-edge and fluorescent screen. It is necessary that the opening in the crystal holder be wider than the incident x-ray beam to prevent scattering by the metal of the crystal holder which may cause a flicker of the milliammeter needle. The G.-M. tube should be set in the housing with the window close to the scatter slit and so positioned horizontally and vertically that the incoming x-ray beam passes along the tube without striking the electrodes.

X-Ray Goniometry of Quartz

The reflection of x-rays by a pile of parallel and equally spaced atomic planes is possible *only* when the Bragg condition is satisfied:

$$n\lambda = 2\,d\,\sin\,\theta, \text{ or}$$

$$\theta = \sin^{-1}\frac{n\lambda}{2d}.$$

When this condition is met, the angle of incidence, θ_i is equal to the angle of reflection θ_r. Therefore, to use a set of atomic planes for reference, their spacing must be known so that the collimated x-ray beam and the Geiger tube can be set at the angle 2θ apart, i.e., the sum of θ_i and θ_r, since the x-ray source is fixed and only the Geiger tube can be set. The

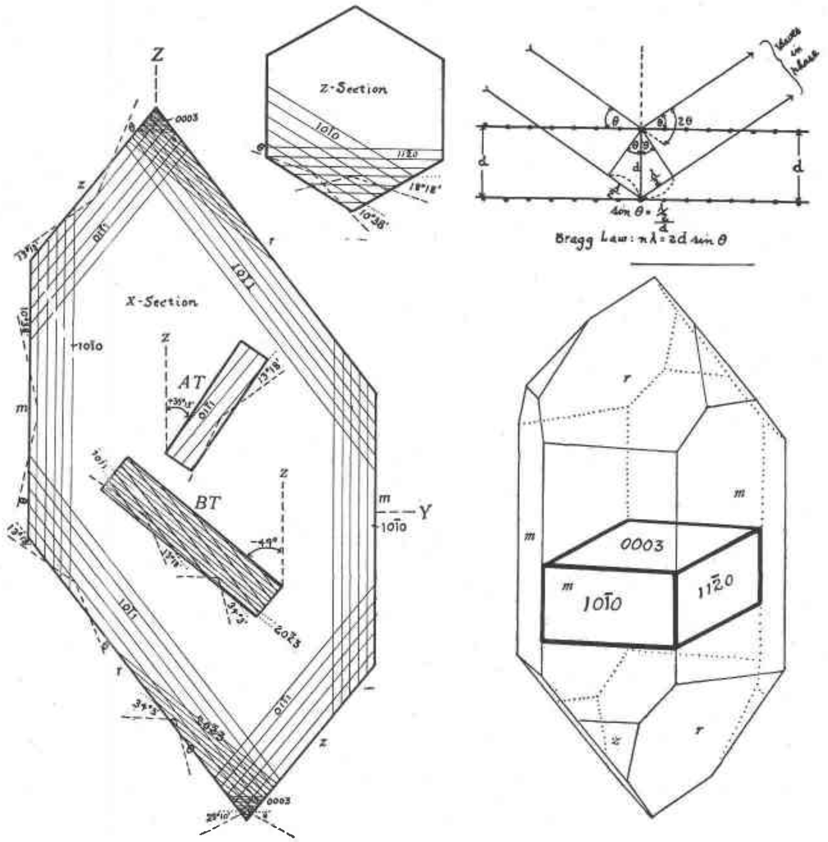


FIG. 9. Commonly used atomic planes for quartz measurement (left). Precisely cut x-ray standards (right) are useful in rapid calibration of x-ray goniometer.

spacing d of any set of atomic planes may be calculated from the precision measurements of the unit cell quoted by Wyckoff[5] using the formula

$$d_{hkl} = \frac{a_o}{\sqrt{4/3\,(h^2+hk+k^2)+\dfrac{l^2}{(c_o/a_o)^2}}}$$

where $a_o = 4.9029$ Å, $c_o = 5.3933$ Å and $h\ k\ l$ refer to the indices of the plane. CuK x-radiation is filtered through 0.0004″ Ni foil to obtain monochromatic Cu$K\alpha$ radiation. The wave-length, λ, is

$$\frac{2\ \text{Cu}\ K\alpha_1 + \text{Cu}\ K\alpha_2}{3} = 1.538674\ \text{Å}.$$

X-ray data for some useful atomic planes of quartz are given in Table 1 and Fig. 9; more complete data are given by Bond and Armstrong.[6]

The x-ray reflections from various sets of atomic planes have different intensities and different natural breadths. For example, reflection from an AT-cut crystal using (01$\bar{1}$1) atomic planes is much stronger and sharper than a reflection from a BT-cut crystal using (20$\bar{2}$3) atomic planes (Fig. 11). The rhombohedral planes are the strongest reflectors in quartz, and in a recorded powder spectrum employing a Geiger tube (Fig. 10) they are 13 on an arbitrary intensity scale in which (20$\bar{2}$3) is 2. The width of the reflection at half the height of finely lapped and etched single quartz crystals cut parallel to the atomic planes, as measured on the Philips crystal analysis unit, is approximately 20′ to 25′ for (10$\bar{1}$1) and 40′ for (02$\bar{2}$3).

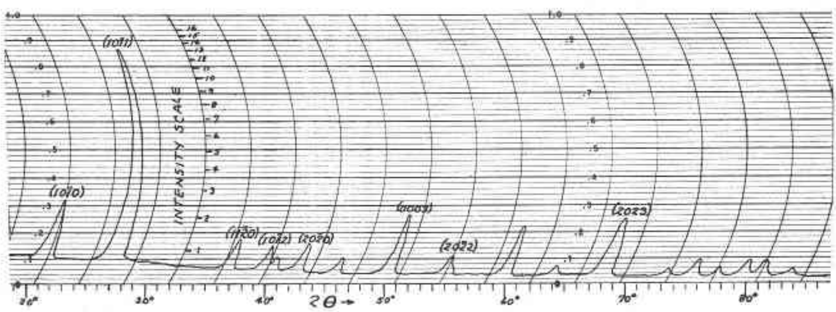


FIG. 10. X-ray powder spectrum of quartz automatically recorded on new Geiger-Muller tube spectrometer. Arbitrary intensity scale. (Prepared by Dr. Herbert Friedman, Naval Research Laboratory.)

Choice of Atomic Reference Planes. It is advisable to choose an atomic plane for reference which is close to parallelism with the surface of the cut. In this way, absorption is reduced to a minimum, reflections are obtained even if the test cut is quite far off angle and errors due to im-

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[5] Wyckoff, Ralph W. G., *The Structure of Crystals*, supplement to 2nd ed., p. 26, Reinhold Publishing Corp., New York (**1935**).

[6] Bond, W. L. and Armstrong, E. J., Use of x-rays for determining the orientation of quartz crystals: *Bell Syst. Tech. Jour.*, **22,** 293–337 (1943).

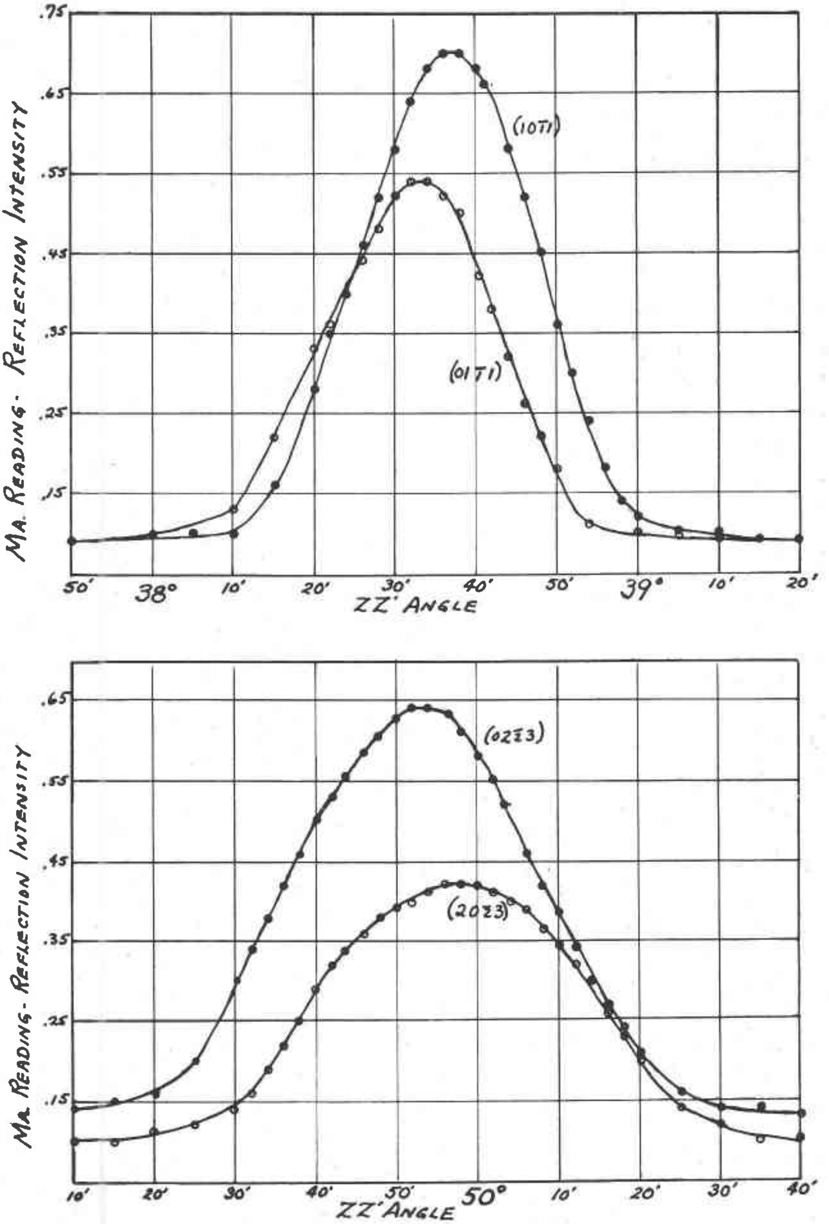

FIG. 11. *X*-ray reflections from electrically twinned etched wafers. Stronger reflection is obtained from (10$\bar{1}$1), the useless side for AT-wafers and from (02$\bar{2}$3) the useless side for BT-wafers. (10$\bar{1}$1) and (01$\bar{1}$1) give much stronger and sharper reflections than (20$\bar{2}$3) and (02$\bar{2}$3). Voltage on G.-M. tube increased for latter to obtain approximately same ma. deflection. Both wafers set in XX′ position. Twins also show deviation of approximately 5′.

proper marking of the X-axis direction are reduced to a minimum. The latter is particularly important in precise measurements of AT- and BT-crystals blanks. For example, if $(10\bar{1}1)$ atomic planes are used to measure the ZZ′ angle of a BT-crystal, the angle between the physical surface of the blank and $(10\bar{1}1)$ will be 49°20′ (nominal)$-$38°13′$=$11°07′. This is the maximum error possible in measuring ZZ′ when the X-axis is 90° from its proper position. Intermediate-errors are obtained for intermediate settings of X. The true ZZ′ angle is measured only when X is vertical and rotation of the blank is about this axis. If $(20\bar{2}3)$ is used for the reference atomic plane for BT-cuts, the error introduced by an incorrect setting is very small; 24′ would be the maximum for a complete 90° error in setting X; in practice, the error in this case is negligible. The commonly used reference planes for the various cuts are listed in Table 1.

X-Ray Standards. Thick crystals (about 0.100″) with marked reference edges cut parallel to the reference atomic planes are useful in setting up and calibrating the *x*-ray goniometer. The crystal is machine lapped and etched to assure freedom from twinning and to sharpen the *x*-ray reflection.[7] The angular-view stauroscope conventions are described in the previous paper by the writers. A line is ruled on the standard to indicate X and an arrowhead to show Z. The following planes are useful for standards: (0003), $(10\bar{1}0)$, $(11\bar{2}0)$, $(10\bar{1}1)$, and $(02\bar{2}3)$. Note that $(10\bar{1}1)$ instead of $(01\bar{1}1)$ and $(02\bar{2}3)$ instead of $(20\bar{2}3)$ are used for standards *only*, to take advantage of the stronger reflections of these planes. It will be apparent that if the standard has been cut exactly parallel to the atomic plane, the *x*-ray reading will be the same for any position of the standard. If the standard is not exactly parallel to the atomic plane, and the X or Z directions are known either from the remains of a natural face or by careful stauroscoping, the exact position on the goniometer scale for that atomic plane may still be determined. One *x*-ray reading is obtained with the arrow up (for rotation about a crystallographic axis), and another from the same surface with the arrow down (180° apart). The atomic plane is half-way between these two readings. For direct settings for AT- and BT-cuts, accurately cut standards at the desired angle to the atomic plane may be cut and X and Z must be marked to determine the correct direction. A standard parallel to the desired atomic plane may also be used by setting the goniometer scale to zero for this reading by rotating the minute arm to the desired ZZ′ angle, and resetting it and the auxiliary scale to this new zero point.

[7] Manning, K. V., The effect of etching on the rocking curve width of calcite crystals: *Phys. Rev.*, **43**, 1050 (1933).

TABLE 1. CRYSTALLOGRAPHIC AND X-RAY DATA FOR QUARTZ

Atomic Plane ($hk\bar{i}l$)	Crystal Face (Dana)	Crystallographic Angles ϕ	ρ	ZZ′	Used for Measuring Plane	Angle Between Plane of Cut and Atomic Plane	G.M. Tube Setting 2θ*	Relative Intensity*
0003	c	00°00′	00°00′	90°00′	Z	0°00′	50°40′	.5[a]
$10\bar{1}3$		"	22°57′	−67°03′			55°22′	45.
$01\bar{1}3$	ω	"	"	+67°03′			"	2.7
$10\bar{1}2$	d	"	32°25′	−57°35′	FT	− 0°35′	39°30′	28.
$01\bar{1}2$	π	"	"	+57°35′			"	85.
$20\bar{2}3$		"	40°16′	−49°44′	BT[b]	+ 0°24′	68°10′	32.
					DT	− 2°14′		
$02\bar{2}3$		"	"	+49°44′	GT[c]	+ 1°46′	"	63.
$10\bar{1}1$	r	"	51°47′	−38°13′	BT[b]	−11°07′	26°40′	100.
$01\bar{1}1$	z	"	"	+38°13′	AT	+ 2°58′	"	75.
					CT	+ 0°13′		
$20\bar{2}2$	r	"	"	−38°13′			54°54′	26.
$02\bar{2}2$	z	"	"	+38°13′			"	26.
$30\bar{3}2$	j	"	62°18′	−27°42′			75°42′	19.
$03\bar{3}2$		"	"	+27°42′			"	27.
$50\bar{5}2$		"	72°31′	−17°29′			143°24′	.25
$05\bar{5}2$		"	"	+17°29′			"	23.
$30\bar{3}1$	M	"	75°18′	−14°42′			68°20′	40.
$03\bar{3}1$	M'	"	"	+14°42′			"	1.4
$10\bar{1}0$	m	"	90°00′	00°00′	Y	0°00′	20°52′	24.
$20\bar{2}0$	m	"	"	"	Y	"	42°28′	28.
$11\bar{2}0$	a	30°00′	"	"	X	"	36°34′	13.[d]

* Data in these two columns from Bond and Armstrong, *Bell Syst. Tech. Jour.*, **22,** p. 303, (1943); these are *estimated* values and do not check well with Figure 11.

[a] This value is in error; (0003) has about the same reflecting power as ($20\bar{2}3$).

[b] Based on ZZ′ = −49°20′.

[c] Based on ZZ′ = +51°30′.

[d] Estimated from Figure 11 by comparison with ($10\bar{1}0$).

The values in brackets (}) in the last column represent x-ray reflection differences useful for determining (+) or (−) angle from Z.

X-RAY MEASUREMENTS OF CUTTING ANGLES

The use of the x-ray technique makes it possible to rapidly and accurately determine the corrections that must be applied to the saw table to bring the cut within the desired tolerance. The following is a description of the most commonly used measurements employed in the manufacture of quartz oscillator-crystals. The marking conventions for correlating the x-ray goniometer readings with saw table corrections are summarized in Table 2.

TABLE 2. MARKING CONVENTIONS FOR CORRELATING X-RAY GONIOMETER AND SAW TABLE

	Horizontal Angle at Saw Table (ZZ′)		Vertical Angle at Saw Table (XX′), AT- or BT-Type Cut	
	AT-Type Cut	BT-Type Cut	Facing Saw, Cut from left side of crystal	Facing Saw, Cut from right side of crystal
Test Cut Wafer	↑	↑	←	→
X-ray Standard or Blank			 or 	 or 

X-Ray Measurement of X-Plane. The desired cut is one parallel to the $(11\bar{2}0)$ atomic planes but the following discussion may be applied to any cut parallel to any atomic plane by simply setting the Geiger tube for that particular reflection.

A vertical arrow pointing upward is marked on the outer surface (side away from the blade) of the test piece to preserve the sense of direction of the cut.[8] Burrs must be removed from the fresh cut surface of the test piece to prevent errors in x-ray measurement. The following discussion refers to both right and left goniometers. By using the terms *clockwise* and *counterclockwise* there is no ambiguity as to direction; it will be noted that clockwise on the right goniometer brings the goniometer arm towards the observer at the front of the machine, and away from him on the left goniometer. The goniometer is set with a $(11\bar{2}0)$ standard so that the scale reads 0°0′ when $(11\bar{2}0)$ atomic planes, $2\theta = 36°34'$ for Cu$K\alpha$, are in a position of maximum reflection.

The test piece is placed flush against the reference face of the x-ray crystal holder with the fresh cut side toward the x-ray beam and the opposite side with the arrow pointing upward is facing the operator. The goniometer arm is rotated clockwise or counterclockewise until the needle of the Geiger tube milliammeter shows a peak reading. If the cut were truly parallel to $(11\bar{2}0)$ the goniometer reading would be 0°0′. In general there will be a deviation, either clockwise or counterclockwise. To show

[8] In some of the earlier x-ray measurements of quartz, while it was appreciated that this sense of direction must be preserved, instead of marking the test cut at the saw, it was stauroscoped. This introduced errors in making the corrections because the stauroscope markings were not necessarily coincident with the axes of rotation of the saw table.

the nature of the correction, let us assume that the peak reading was at 1°35′ clockwise from 0° on the scale. At 0° the test cut was *not* parallel to $(11\bar{2}0)$ and it was necessary to correct the setting by 1°35′ clockwise to bring it parallel to the $(11\bar{2}0)$ planes. It is apparent that the quartz on the saw table corresponds to the first position at the x-ray goniometer, 0°, and that it is therefore necessary to make exactly the same correction, in the same direction, 1°35′ clockwise, at the saw. It is also apparent that there has been no change in position of the quartz or the test cuts between the saw and the x-ray apparatus as is evinced by the procedure of marking the vertical arrow on the test cut, and preserving this direction on the x-ray crystal holder.

To test the vertical angle of the saw table, the test piece is rotated 90° so that the arrow is now horizontal and another position of maximum reflection obtained. If the test cut has been made from the right side of the crystal (when facing the saw) the arrow points to the right; if the test cut was made from the left side of the crystal, the arrow should be pointed to the left to obtain a direct reading of the correction that must be applied to the vertical plane of the saw table. An alternative procedure is to point the arrow in the same direction for either right- or left-side test cuts and correct the former in the same direction and the latter in the opposite direction to that indicated by the x-ray reading.

X-Ray Measurement of AT-Test Cuts. AT-wafers are cut at an angle of +35°15′ from the optic axis, or at an angle of −2°58′ from the z $(01\bar{1}1)$ plane which is at +38°13′ from Z. It is therefore convenient to check the ZZ′ angle (horizontal angle of the saw table) of cut with $(01\bar{1}1)$; i.e., the plane of the sawed surface should be −2°58′ from the $(01\bar{1}1)$ atomic reference planes within the quartz wafer. It will be obvious that x-rays cannot distinguish between a plane cut at −2°58′ (ZZ′=35°15′) from one cut at +2°58′ (ZZ′=41°11′) to z $(01\bar{1}1)$ so that it is essential to transfer the sense of the cut from the saw to the x-ray machine.[9]

The most convenient procedure is to preset the goniometer by means of either a precisely cut AT or $(10\bar{1}1)$ atomic plane standard as described in the section on x-ray standards. If an AT-standard is used it should be positioned with the line indicating the electric axis vertical and the arrowhead indicating the optic axis direction to the left as determined with the angular view stauroscope. (The stauroscope markings on the standard are not to be confused with the markings of the test cut.) Test wafers, like all other test cuts, are marked at the saw table with a vertical arrow

[9] One way of distinguishing them is to set the goniometer arm to zero for $(20\bar{2}3)$, ZZ′=49°44′. If the cut is at +2°58′ from $(01\bar{1}1)$, a reflection will be obtained at 8°33′ or at 12°59′ if cut at −2°58′ when the X-axis is vertical. For reasons mentioned above, an atomic reference plane closer to the surface is required for precision measurements.

pointed upward on the side of the test wafer opposite the fresh cut. The wafer is set in the crystal holder so that the x-ray beam strikes the fresh cut surface with the arrow in view of the operator. The arrow is pointed upward to measure the correction required for the horizontal plane (ZZ′) of the saw table. If the goniometer reading is not 0° (corresponding with the x-ray standard setting) an angular correction of the same amount and in the same direction, clockwise or counterclockwise, is marked on the wafer to be duplicated at the saw.

Since the surface of the wafer is inclined to the atomic reference planes, the horizontal and vertical angle corrections are read from different points of the x-ray goniometer scale. Thus XX′ 38°13′−ZZ′ 35°15′= 2°58′ apart for AT-cuts.[10] To measure the vertical angle, XX′, the goniometer is set by means of an x-ray standard with its zero point corresponding to 38°13′ for maximum reflection from $(01\bar{1}1)$. In this setting the arrow is horizontal and if XX′=0°, the atomic planes are parallel to the edge of the wafer. The arrow is pointed to the right if the test cut was made from the right side of the crystal, and to the left, if cut from the left side, to obtain a direct reading of the vertical angle correction and direction that must be duplicated at the saw table.

X-Ray Measurement of BT-Test Cuts. The turning point of the temperature-frequency curve varies with ZZ′ angle and in BT crystals, angles varying from −49°00′ to −49°30′ are used, depending upon the desired properties. For this discussion we will assume a selected angle of −49° 20′. BT-wafers are cut at an angle of +11°07′ from the r $(10\bar{1}1)$ major rhomb face which is at −38°13′ from Z. As described above, it is desirable to use an atomic reference plane close to parallelism with the cutting surface. The best planes to use are $(20\bar{2}3)$ which are but +24′ from the plane of the cut $(Z\wedge(20\bar{2}3)=49°44')$.

Horizontal and vertical angular measurements and corrections are made in the same way as described above for AT-wafers, with these two exceptions: (1) the BT or $(02\bar{2}3)$ standard is placed in the x-ray crystal holder with X-axis vertical and optic axis direction to the *right;* (2) the vertical angle reading, XX′, is made 24′ from the ZZ′ reading.

X-Ray Measurement of AT- and BT-Wafers and Blanks. It is often necessary to check the angles of a stock of blanks or wafers to determine ZZ′ and XX′. X-ray measurements of all blanks prior to lapping is part of the inspection scheme to eliminate those not meeting the rigid angular specifications. Although x-ray measurements are used to control the cutting angles, errors creep in due to drift of the blade, inaccurate ways of the saw, etc. The x-ray measurements are preceded by orientation of

[10] Where large production is required it is advisable to set up one goniometer to measure ZZ′ directly and the other XX′.

the blank or wafer in the angular-view stauroscope, by means of which the optic axis direction, indicated by an arrowhead, and the direction of the electric axis, indicated by a line, are identified. This stauroscope pre-orientation permits setting of the blank in the *x*-ray beam in a position comparable to its orientation in the quartz when it was cut and avoids all ambiguity as to whether XX′ or ZZ′ is being measured or if the reading is "high" or "low." Blanks cut from wafers with reference edges (X-planes in the X-block method) and laid out by means of a rubber stamp with marked stauroscope directions need no further stauroscoping. Furthermore, if they have been properly squared the edges of the blank are parallel to X and Z′.

AT-cuts may be checked by reference to the $(01\bar{1}1)$ atomic planes with the Geiger tube set at 26°40′. The ZZ′ angle is measured by placing the blank in the holder with the arrow-head (Z direction) pointing to the *left* (marked side of blank toward operator and away from *x*-ray beam); rotation is thus about the X-axis. The XX′ angle is measured by turning the blank 90° with the arrowhead pointed either up or down.

BT-cuts are checked by reference to the $(20\bar{2}3)$ atomic planes with the Geiger tube set at 68°10′. The ZZ′ angle is measured by placing the blank in the holder with the arrow-head (Z direction) pointing to the *right*. XX′ is measured with the arrowhead pointed either up or down.

The *x*-ray goniometer is accurately preset to 0°0′ with an *x*-ray standard and must be reset or read from different points of the scale when switching from ZZ′ to XX′ measurements as described above.

Precise Angular Adjustments with *X*-Rays

In addition to being an invaluable technique for measuring angles of cuts, *x*-rays are used to adjust cut wafers on jigs for angle correction by lapping when required; for determining the error in crystals set up with the mounting stauroscope prior to cutting so that the saw can be adjusted before making a test cut; and in the precise location of X- and Y-axes in Z-sections approximately oriented by other methods.

X-Ray Adjustment of Wafer Angle Correction Jigs. This procedure was introduced to correct wafers cut by the flat-lay method. Its purpose is to bring a wafer to adjustment so that its surface can be ground to the proper plane. The wafers are individually cemented to a tilting plate on one end of a rather expensive, specially designed, cylindrical jig, with a chamfered edge of the wafer parallel to two of the four adjusting screws at the other end of the cylinder. Each pair of screws tilts the wafer at right angles to the other pair. The jig is set in a special preset chuck in the *x*-ray beam and the wafer adjusted until *x*-ray reflection shows that the plane of cut desired is exactly perpendicular to the axis of the cylinder.

The jig is then rotated 90° and the adjustment repeated. A number of jigs are locked to the rotating arm of an ultralap which quickly trues the surface. The wafers are then recemented with the trued face down and the other side corrected with the ultralap.

X-Ray Presetting of Mounted Quartz Crystals. In large scale production, where every extra cut soon involves enormous additional costs, every attempt must be made to reduce the number of test cuts. This can be effectively done by *x*-ray measurement of the accuracy of the mounting and subsequent correction of the saw table before the first test cut is made. This does not eliminate the necessity of making test cuts, because in actual practice it is extremely difficult to preset within the precision usually required, but there is better chance for the accurate adjustment of the crystal for cutting with only one test cut.

The quartz crystals are mounted on a prism or rhombohedral (major or minor) face with the optic axis parallel to the long reference edge of a rectangular glass plate, utilizing the mounting stauroscope. The mount is set in the *x*-ray beam with the short reference edge of the glass in contact with the reference bar of the *x*-ray platform (Fig. 6). With the Geiger tube set at 36°34′ for reflection from $(11\bar{2}0)$ planes, the goniometer arm is rotated to a position of maximum reflection. If the goniometer scale has been preset to 0° by means of a precisely cut standard, the deviation of the optic axis of the quartz with respect to the short reference edge of the glass plate can be read directly. The angular correction, and its clockwise or counterclockwise direction, are marked on the glass plate so that the setting may be duplicated at the saw.

The same procedure may be followed in presetting X-blocks prior to wafering, by using the $(10\bar{1}0)$ atomic planes and in setting up Z-sections prior to cutting X-blocks or Z-bars.

Precise Location of X- and Y-Axes in Z-Sections. In cutting large quartz crystals, the strategy calls for the preliminary sawing into Z-sections, and then into X-Sections (or if the quartz has large flawless areas, into Y-bars). The general direction of the X- or Y-axes can be determined from the light figures of the etched Z-sections, by optical twinning if present, or by the other methods described. An accurate location may then be obtained by *x*-rays. The Z-section is placed on a platform (Fig. 5) on the *x*-ray goniometer with the approximate direction determined from the light figure in the approximate reflection position. The goniometer arm (and platform) is rotated until a peak milliammeter reading indicates the desired atomic planes are in the position of maximum reflection. This direction is ruled on the section by means of a right angle marking guide previously set with a standard to determine X with $(10\bar{1}0)$ or Y with

$(11\bar{2}0)$. Since these two axes are perpendicular to each other, the right angle permits marking one axis if the other cannot be found due to irregularity of the edge of the Z-section.

X-RAY APPLICATIONS USING REFLECTION INTENSITY DIFFERENCES

The marked differences of reflecting power of certain atomic planes on either side of the optic axis has made it possible to apply x-rays to the determination of usable portions of electrically twinned wafers, and the negative or positive cutting directions from Z. These methods are not widely used because simpler tests more adaptable to production routine are available.

X-Ray Determination of Usable Parts of Electrically Twinned Wafers. A test for the usable portion of electrically twinned wafers can be readily made by x-rays.[11] It is based on the difference in intensity of reflection of x-rays from r $(10\bar{1}1)$ and z $(01\bar{1}1)$ planes. The etched AT-wafer is set in the x-ray beam and the goniometer adjusted to a peak reading. The micro-adjustment of the voltage on the Geiger tube is set so the reading is at approximately mid-scale on the milliammeter. The shutter is closed and the wafer pushed horizontally to bring an area on the opposite side of the twin boundary into the x-ray beam. The intensity of reflection will change and be higher if on the side of the twin boundary reflecting from r $(10\bar{1}1)$ as shown in Fig. 10. The test is also applicable to etched BT-wafers with the Geiger tube set at 68°10′ for reflection from $(20\bar{2}3)$. In this case the lower reflection is from the side nearly parallel to the major rhomb. Thus in AT- and BT-wafers, that portion of the electrical twin with the weaker x-ray reflection is the usable side. The position of the two individuals is also shifted 2′ to 5′.

X-Ray Method of Determining Negative and Positive Directions from Z. This method was an early one for this purpose, and depends upon the fact that the $(30\bar{3}1)$ reflects strongly but $(03\bar{3}1)$ does not reflect at all.[12] The Geiger tube is set at 68°20′ and a wedge of 14°42′ is used to tilt an untwinned etched portion of the Z-section so that $(30\bar{3}1)$ is vertical. If no reflection is obtained, the Z-section is turned upside down on the wedge platform.

[11] This method was developed by Dr. Dominick D'Eustachio, "A method for determining which portion of a twinned wafer should be used": *Tech. News Bull. No. 6*, Signal Corps Ground Signal Agency (1943).

[12] This reflection asymmetry was pointed out by R. E. Gibbs, Structure of quartz: *Proc. Roy. Soc. (London)*, **110,** 443–455 (1926). See also Broughton, William W., Piezoelectric apparatus: *U. S. Patent Off.* No. 2,151,736, Mar. 28, 1939 and Bond, W. L. and Armstrong, E. J., *op. cit.*

Possible Applications in Other Fields

The x-ray techniques described here may be developed for application in other fields. It has been used in the cutting of calcite and small diamond crystals for wire dies have been oriented with the technique. The orientation of sapphire for jewel bearings and other applications seem apparent.

The use of the G.-M. tube for diffraction work is a field which deserves the attention of x-ray crystallographers.[13]

[13] Friedman, H., Geiger-counter spectrometer for industrial research: *Electronics*, **18,** 132–137 (1945).