ORIENTATION TECHNIQUES FOR THE MANUFACTURE OF QUARTZ OSCILLATOR-PLATES

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CONTENTS

ABSTRACT

This paper describes the theory, methods and equipment used in orienting raw quartz, and the reorientation of sections, bars, wafers and blanks in various stages of processing. The orientation of quartz by optical determination of its "hand" and electrical measurement of polarity of the electric axis is discouraged because the latter test may be completely misleading due to electrical twinning. The characteristic light figures, obtained on etched sections and bars when placed over an illuminated pin-hole, are the sole infallible and unambiguous criteria for the orientation of quartz. The light figures on etched Z- and X-sections and the determination of usable portions of electrical twins in etched wafers by characteristic light reflection or transmission are described. The optical methods include the use of two simple types of stauroscopes: one for orienting Z parallel to the reference edge of a glass mounting plate, and the other for determining X and Z directions in wafers. The conoscope is valuable for locating Z in quartz whether faced or defaced. The notation

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of axes, faces and cuts is given; all ambiguity is removed by referring the cuts to the crystal faces. Various minor methods such as the development of parting parallel to the X-axes by slowly immersing a heated thin basal section in water are reviewed.

INTRODUCTION

The quartz oscillator, as used for frequency control in radio and telephone communication systems, is one of the most precise devices developed by modern science.[1] Resembling small square microscope cover glasses, plates of one frequency may differ in thickness from plates of adjoining channels by as little as one ten-millionth of an inch. Theoretically, a difference of ten cycles, which is easily measurable, means a reduction in thickness of but one layer approximating a unit cell in thickness, in the case of an 8 Mc/sec BT crystal.

The quartz plates used by MM. Jacques and Pierre Curie in their discovery of the piezoelectric effect were "X-cuts" with their largest surface perpendicular to the electric axis, X (the crystallographic axis *a*), length parallel to the mechanical axis, Y, and width parallel to the optic axis, Z (crystallographic axis *c*).[2] Such a plate, parallel to $(11\bar{2}0)$, could be cut by simply laying a crystal on its prism face (m $10\bar{1}0$), and sawing parallel to the vertical optic axis (Z, *c*); it was not necessary to determine the hand of the crystal or polarity of the X-axis.

Although X-cuts are still used in ultrasonic devices and other special applications where frequency change with temperature is of no importance, or may be controlled by the use of thermostatically controlled ovens, the vast majority of the crystals manufactured today are of the low temperature coefficient type.[3] These crystals are cut at selected pre-

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[1] In a paper by C. F. Booth, The application and use of quartz crystals in telecommunications: *Jour. Inst. Elect. Eng.*, **88,** 97–144 (1941), Mr. A. J. Gill states (p. 129) "the author mentions that the error of a certain crystal standard is ±0.00086 sec. in 24 hours; this means that a clock driven from this frequency standard would gain or lose less than 1 sec. in 3 years, and in this period the crystal will have made 10^{14} vibrations."

[2] Curie, Jacques et Pierre, Developpement, par pression, de l'electricité polaire dans les cristaux hemiedres à faces inclinées: *Compt. Rend.* **91,** 294–295 (1880); Sur l'electricité polaire dans le cristaux hemiedres à faces inclinées: *ibid.*, **91,** 383–386 (1880); Contractions et dilatations produites par les tensions electriques dans les cristaux hemiedres à faces inclinées: *ibid.*, **93,** 1137–1140 (1881). Friedel, C., and Curie, J., Sur la pyroélectricité du quartz: *Bull. Soc. franc. Min.*, **5,** 282–296 (1882).

[3] The Coast Guard desiring to use quartz crystals to control radio communications from the cold of Alaska to the warmth of Florida requested more stable plates from the Western Electric Co. a little more than a decade ago.

More than 95% of the crystals manufactured today are AT-(1 to 5.5 MC), BT-(5.5 to 9 MC) and CT-(200 to 500 KC) cuts. For a description of the properties of these and other types of crystals see: Mason, W. P., Low temperature coefficient crystals: *Bell System Tech. Jour.*, **19,** 74–93 (1940); Lack, F. R., Willard, G. I. and Fair, I. E., Some improvecise angles to the optic axis but with one edge parallel to the electric axis (except the GT-cut) and do not require temperature control to keep military communications within their specified channels. The most commonly used cuts and their orientations are shown in Fig. 1. Temperature control is required only where accuracy of the order of a few parts per million must be maintained as in commercial broadcast stations, frequency standards, etc.

Radio engineers defined the cuts with respect to the electrical axis (X) and the optic axis (Z). A quartz crystal was squeezed between two opposite prism edges (from which the X-axis emerges) and the reaction noted on an electrometer or similar device. If the polarity of the edge under test was positive on compression, the AT-cut was made at an angle of +35°15′ (clockwise) from the optic axis in a right-hand crystal, and the BT-cut at an angle of −49° (counterclockwise) from the optic axis. The directions were reversed for a left-hand crystal. A polariscope was used to determine the hand of the crystal.

The usage of the terms right- and left-hand by radio engineers has been generally the *reverse* of that of crystallographers and mineralogists. In using the Herschel convention the radio terminology has followed that employed by chemists in polarimetry and saccharimetry in which a quartz crystal is called right-handed if it rotates the plane of polarization to the right (clockwise) when the eye is following the light rays. The confusion has arisen from the apparent reversal of the direction when observed with the eye adjacent to the crystal looking toward the oncoming rays (Biot convention). A discussion of the confusion in the nomenclature of rotatory power is given by Sosman.[4] All ambiguity is removed if the cuts are related to the crystal faces.[5]

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ments in quartz crystal circuit elements: *ibid.*, **13**, 453–463 (1934). Low temperature coefficient crystals were also developed independently at about the same time in Germany and Japan: Bechmann, R., Temperature coefficients of vibrations of quartz plates and bars: *Hochfreq. Tech. u. El. Ak.*, **44**, 145–160 (1934); Straubel, J., *Zeit. tech. Physik.*, **35**, 179 (1934); Koga, I., Thermal characteristics of piezoelectric oscillating quartz plates: *Report of Radio Researches and Works in Japan*, **4**, 61 (1934). See also series of papers in *Bell Syst. Tech. Jour.*, **22** (1943), **23** (1944) and *Bell Lab. Record*, **22** (1943–44).

The GT-cut crystal, ZZ′ = +51°30′ with its edge rotated 45° from the X-axis, has an extraordinarily low temperature coefficient. Used for low frequencies (100 KC), it is constant to one part in a million in the temperature range 0° to 100°C. and over a 30°C. region the frequency change is one part in ten million with no temperature control. Mason, W. P., A new quartz crystal plate, designated the GT, which produces a very constant frequency over a wide temperature range: *Proc. Inst. Radio Eng.*, **28**, 220–223 (1940).

[4] Sosman, Robert B., *The Properties of Silica*, The Chemical Catalog Co., Inc., New York (**1927**), pp. 648–649. *Dana's Textbook* and *Manual* use the Herschel convention in the optical parts of the books but the Biot convention is followed in the crystallographic

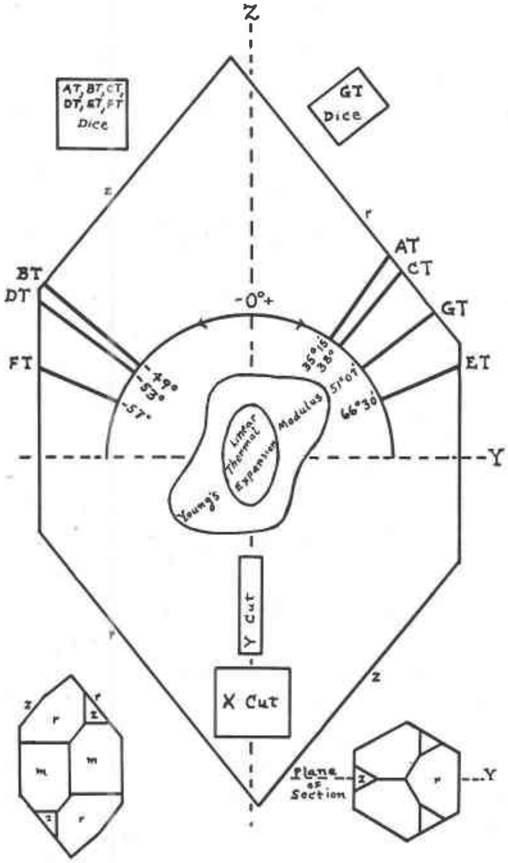

FIG. 1

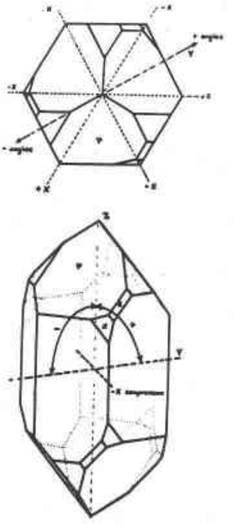

FIG. 2

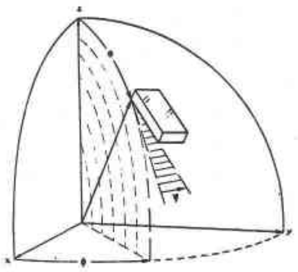

FIG. 3

FIG. 1. Orientation of principal types of oscillator-crystals and their cutting angles. AT, CT, GT and ET approximately parallel to minor rhomb *z;* BT, DT and FT approximately parallel to major rhomb *r*. X parallel to edge of plates except GT which is rotated 45°. X- and Y-cuts are perpendicular to X- and Y-axes, respectively.

FIG. 2. The principal axes of quartz. $Z=c=$optic axis; $X=a=$electric axes; $Y=$mechanical axes. (+) direction from Z is rotation towards parallelism with *z* and (−) direction towards parallelism with *r* regardless of hand of crystal. (+) is clockwise in right-hand crystal and (−) counterclockwise but are reversed in left-hand crystal. In an untwinned crystal the ends of the X-axis on a prism edge containing the *s* and *x* faces become negatively charged on compression and positively charged on release of compression (tension or stretching) by application of a stress along X.

FIG. 3. I.R.E. Orientation angles. ϕ measured from X $(a)=$complement of crystallographer's ϕ; θ measured from Z $(c)=\rho$ of crystallographers; $\psi=$dicing angle. Drawing from W. L. Bond, *Bell Syst. Tech. Jour.*, **22,** 224 (1943).

The manufacture of quartz oscillator-plates involves not only the orientation of the "raw" quartz, usually known as "mothers" in the industry—whether faced or completely defaced, but the reorientation of sections, bars, wafers, or blanks in various stages of processing, and the recognition of electrical and optical twinning in this material—and particularly the orientation of the latter in the wafers under examination. Moreover, the angular deviations from specified tolerances must be accurately determined and controlled. While this seems a formidable task, it may be readily accomplished from the morphology of the quartz by the unique properties along the optic axis, the etch patterns shown in various sections, or by measurement of angles from reference atomic planes within the crystal by means of x-rays. The use of the x-ray method requires prior orientation by some of the methods described here, before it can be successfully applied, and is described in the following paper.[6]

This paper deals only with certain technological aspects of the practical problems closely related to the manufacture of quartz oscillator-plates. A portion of this subject matter especially prepared for crystal manufacturers has been published by the Signal Corps.[7] Time and space requirements do not permit the presentation of crystallographic data collected on etch figures, x-ray reflections, inclusions and a host of other interesting phenomena. For the same reasons, the literature cited is not a complete bibliography. It is our desire to present these supplementary data at the earliest possible date.

Acknowledgments. The writers are indebted to Mr. John Derbyshire, N.A.P., for taking many of the photographs and Mr. Charles E. Goldmann, N.A.P., for converting the stauroscopes illustrated, from commercial instruments. The conoscope was designed by Mr. W. L. Bond of Bell Telephone Laboratories. Figures 3 and 12 are from Bond, *Bell*

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description of quartz. Other inconsistencies have been pointed out by K. S. Van Dyke, On the right- and left-handedness of quartz and its relation to elastic and other properties: *Proc. Inst. Radio Eng.*, **28,** 399–406 (1940). The adaption of the Biot convention which makes a given crystal either right- or left-handed in both the commonly used crystallographic and optical descriptions is recommended also to radio engineers by W. G. Cady and K. S. Van Dyke, Proposed standard conventions for expressing the elastic and piezoelectric properties of right and left quartz: *ibid.*, **30,** 495–499 (1942).

[5] The Japanese have defined cuts by their angles from the major and minor rhombohedral faces.

[6] Parrish, William and Gordon, Samuel G., Precise angular control of quartz cutting with x-rays: *Am. Mineral.*, this issue.

[7] Parrish, William and Gordon, Samuel G., *Manual for the Manufacture of Quartz Oscillator-Blanks,* Office of the Chief Signal Officer, War Department, Washington, D.C., 1943. Note: this publication is no longer classified as *restricted.*

Syst. Tech. Jour., **22,** 224 (1943). Figure 4 showing the Airy's spirals is from C. F. Booth, *Jour. Inst. Elect. Eng.*, **88,** 97 (1941). Mr. John D. Davies, N.A.P., aided in preparation of the section on electrical polarity determination.

Notation of Axes, Faces, and Cuts

The optic axis (crystallographic axis c) is conventionally designated Z. The horizontal crystallographic axes a_1, a_2, and a_3, are called the electric axes, each designated by the letter X. The three intermediary, horizontal axes, each at right angles to an X axis, are called the Y axes, or mechanical axes (Fig. 2).

The striations on the prism faces m $(10\bar{1}0)$ are parallel to the X axes, which emerge at the prism edges (normals to $11\bar{2}0$). The normals to the prism faces m $(10\bar{1}0)$ are the Y axes. Due to the oscillatory development of the prism faces (between the prism and terminal rhombohedrons), the prism edges are but rarely parallel to the optic axis.

The terminal rhombohedrons, r $(10\bar{1}1)$ and z $(01\bar{1}1)$ are distinguished as the *major* and *minor* rhomb faces, respectively.

The modifying planes s $(11\bar{2}1)$ the right trigonal pyramid, and x $(51\bar{6}1)$ the right positive trapezohedron, form a zone with m $(10\bar{1}0)$ and z $(01\bar{1}1)$, to the right of m $(10\bar{1}0)$ and serve to identify such a crystal as right-handed (Fig. 5). The left trigonal prism $'s$ $(2\bar{1}\bar{1}1)$ and the left positive trapezohedron $'x$ $(6\bar{1}\bar{5}1)$ form a zone with m and z to the left in left-hand crystals (Fig. 4).

A right-hand crystal appears to rotate the plane of polarization to the right (clockwise) on looking towards the light source (Biot convention).

It will be noted that there are six major rhombohedral faces (r), three at each end of the crystal; and likewise six minor rhombohedral faces (z) at each end of the crystal. For each r face, there is another r face—opposite and parallel to it on the other end of the crystal. Likewise, each z face is opposite and parallel to another z face. Thus there are three pairs of parallel major (r) rhomb faces, and between them three pairs of minor (z) rhomb faces.

The AT-, CT-, ET-, and GT-cuts may be thought of as made between a pair of minor (z) rhomb faces, and not far in angle from parallelism with face z.

The BT-, DT-, and FT-cuts may be considered as made between a pair of major (r) rhomb faces, and not far in angle from parallelism with face r.

The "I.R.E. Orientation Angles." A committee of the Institute of Radio Engineers has recommended the adoption of the following method of specifying the orientation of a quartz plate. In this new system, a cut is defined with respect to three axes at right angles to each other: Z (the *c* or optic axis), X, the electric axis (the *a* axis normal to $11\bar{2}0$), and Y, the mechanical axis (normal to *m* $10\bar{1}0$) as shown in Fig. 3. It will seem strange to crystallographers that three angles are necessary to define a

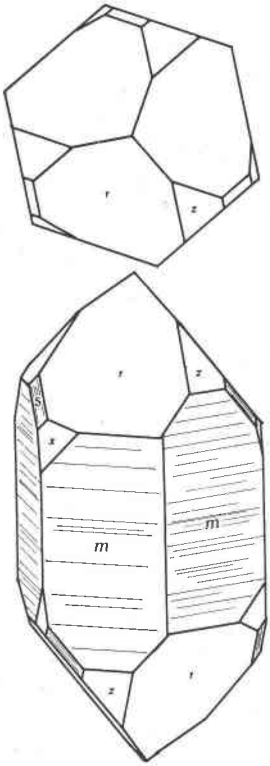

FIG. 4

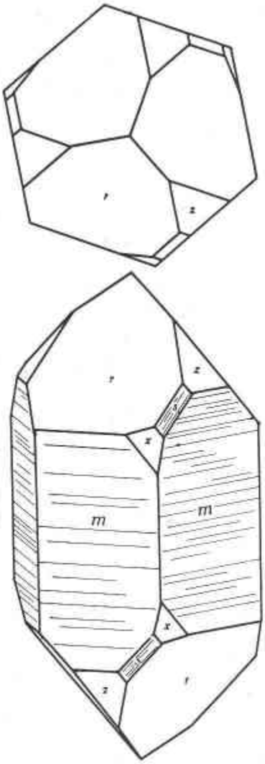

FIG. 5

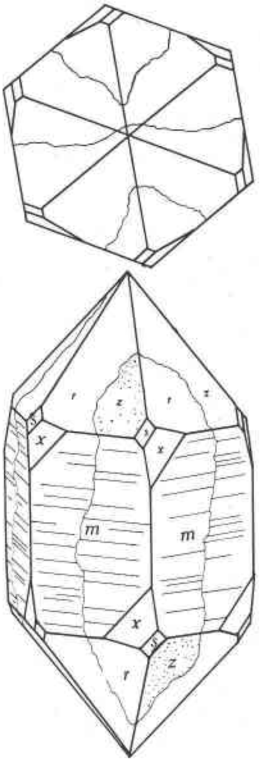

FIG. 6

FIG. 4. Left-hand quartz. With Z (*c*) vertical, *'s* $(2\bar{1}\bar{1}1)$ and *'x* $(6\bar{1}\bar{5}1)$ form a zone with *m* $(10\bar{1}0)$ and *z* $(01\bar{1}1)$ to the *left* of *m*. Rotation of plane of polarization is to the *left, counterclockwise,* on looking *toward* source of light (Biot convention).

FIG. 5. Right-hand quartz. With Z (*c*) vertical, *s* $(11\bar{2}1)$ and *x* $(51\bar{6}1)$ form a zone with *m* and *z* to the right of *m*. Rotation of plane of polarization to the *right, clockwise* (Biot convention).

FIG. 6. Electrical twinning (Dauphiné law) of left-hand individuals. The individuals are of the same hand and related by a 180° rotation about Z (*c*) so that the crystallographic axes are parallel but the polarities of the electric axes X (*a*) are reversed and *r* and *z* become coplanar. Electric polarity determination of front edge of crystal would be misleading for orientation purposes due to electrical twinning.

plane but this is true of only two of the angles ϕ and θ. ϕ is measured from the electric axis (crystal axis a) and is therefore the complement of ϕ of crystallographers. The angle θ corresponds to the ρ angle of crystallography. The third angle ψ, is merely the dicing angle, the angle which the long edge of the blank makes with the trace of the meridian ϕ.

Cut[8]	ϕ	θ	ψ
z	0°	0°	0°
x	0	90	90
y	90	90	90
AT	−90	$54\frac{3}{4}$	90
BT	−90	−41	90
CT	−90	52	90
GT	−90	38°52′	±45
NT	6°40′	50°28′	79°36′

Radio engineers often refer to the ZZ′ and XX′ angles. ZZ′ is the angle between the optic axis, Z, and the plane of the blank, Z′. XX′ is the angle between the electric axis, X, and the edge of the blank, X′.

ELECTRICAL DETERMINATION OF POLARITY OF X-AXIS

The polarity of the electric axis X may be determined by applying pressure across opposite prism edges and noting the polarity of the voltage produced with a suitable electronic circuit. In quartz it is a rapid *change* in pressure which produces the voltage. The polarity on tension (release of pressure) is the reverse of that obtained on compression. *In an untwinned left- or right-hand crystal, that end of the X-axis which emerges at a prism edge below the modifying s and x faces becomes negatively charged on compression.* This is consistent with the kind of charges developed on this edge on *cooling* a quartz crystal.[9] Here the temperature *gradient* is due to the inside of the crystal being at a higher temperature than the surface. The temperature gradient is reversed on heating the crystal and the charges developed have the opposite sign.[10] The pyroelectric test may be tried by Kundt's method using charged red lead and sulfur pow-

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[8] Bond, W. L., Method for specifying quartz crystal orientation and their determination by optical means: *Bell Syst. Tech. Jour.*, **22,** 224–262 (1943).

[9] Von Kolenko, B., Die Pyro-Elektricitat des Quarzes in Besuch aus sein kristallographisches System: *Zeit. Kryst.*, **9,** 1–28 (1884).

[10] Wooster, W. A., *A Text Book on Crystal Physics*, Cambridge University Press, Macmillan, New York (**1938**), Chap. VII, Pyro-Electricity.

ders[11] or the magnesium smoke[12] or liquid air methods[13] but has not been employed as a production technique in this country.

Several types of devices have been developed to determine the piezoelectric effect. Where quantitative measurements are not required the polarity indicator shown in Fig. 7 may be employed. It gives the polarity of the X-axis which emerges at the edge which is *down* on the lower platform. A rapid *change* in pressure produces a voltage which is impressed on the grid of a high gain amplifier such as the 6J7. A negative charge impressed on the grid will decrease the plate current and a positive charge will cause an increase in plate current. A milliammeter in the plate circuit of the 6J7 permits visual indication of the polarity. The plate current can be adjusted to one-half full scale value on the meter thus allowing + or − readings on the same meter. The meter deflection is observed immediately on applying pressure by means of the arm; it is reversed on releasing pressure.[14]

ORIENTATION BY MORPHOLOGY

In a well developed crystal it is easy to recognize the optic axis, (*c*, Z) which coincides with the principal crystallographic axis; the electric axes (X) $[11\bar{2}0]$ which are also the striations on *m* $(10\bar{1}0)$ and the mechanical axes (Y) which are the normals to the prism faces (*m* $10\bar{1}0$).

More difficult, however, is the distinction between the rhombohedral planes *r* $(10\bar{1}1)$, the major rhomb; and *z* $(01\bar{1}1)$, the minor rhomb; and since Dauphiné (electrical) twinning, in which *r* and *z* are coplanar is so common, the effort is futile. Much faith has been placed on determinations based upon the relative size of the *r* and *z* faces, and in the general rule that in "candles" the prism faces become narrower below *r* faces. Where triangular etch pits occur on the prism faces, the apices of the triangles always point to the major (*r* $10\bar{1}1$) faces (Fig. 8). Striations when discernible on *s* $(11\bar{2}1)$ are parallel to the major rhomb *r* $(10\bar{1}1)$ (Fig. 8). Crystals which have adjacent terminal faces of the same size

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[11] Kundt, A., Ueber eine einfach Methode zur Untersuchung der Thermo-, Actino- und Piezoelectricitat der Krystalle: *Ann. Phys. u. Chem.*, **20**, 592–601 (1883).

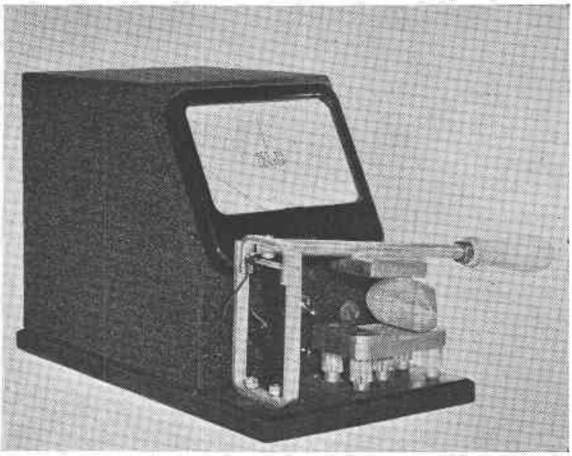
[12] Maurice, M. E. On the demonstration of electric lines of force, and a new method of measuring the electric moment of tourmaline: *Proc. Camb. Phil. Soc.*, **26**, 491–495 (1930).

[13] Bleekrode, L., Über einige Versuche mit flüssiger Luft: *Ann. Phys.*, **12**, 218–223 (1903). Martin, A. J. P., On a new method of detecting pyro-electricity: *Mineral. Mag.*, **22**, 519–523 (1931).

[14] Electrical twinning in unetched quartz sections was once determined with an apparatus for detecting polarity with a probing electrode. A grid was ruled on the section, or illuminated from below, the surface was probed and the resulting polarities contoured on the grid!

and those in which the edges of adjacent terminal faces are parallel are almost certainly twin crystals.

The zone of minor faces *x* $(51\bar{6}1)$, *s* $(11\bar{2}1)$ and *z* $(01\bar{1}1)$ are to the right of *r* $(10\bar{1}1)$ in a right-hand crystal, and to the left in a left-hand crystal.



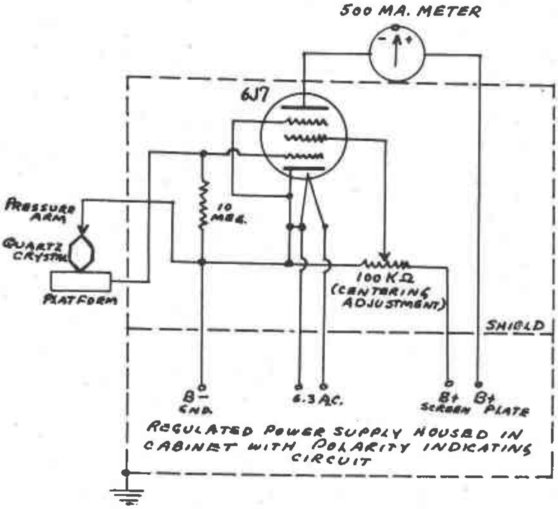


FIG. 7a. Piezometer for determining polarity of electric axes by squeezing crystal along X and noting meter deflection. Reaction must be noted immediately on squeezing and reverses on release of pressure. Fig. 7b. Circuit prepared by John D. Davies, NAP.

It is therefore necessary to supplement this observation with an optical test for chirality. Even if these faces are absent, the prism edge which would have been contiguous to them gives a negative reaction on compression on the piezometer, and thus serves as an aid in orientation. It must be emphasized that such a test is diagnostic only for the point squeezed, and if the quartz is electrically twinned, is ambiguous. If an electrically twinned crystal such as the one shown in Fig. 6 was oriented by testing the front side, the resulting wafers would be useless.

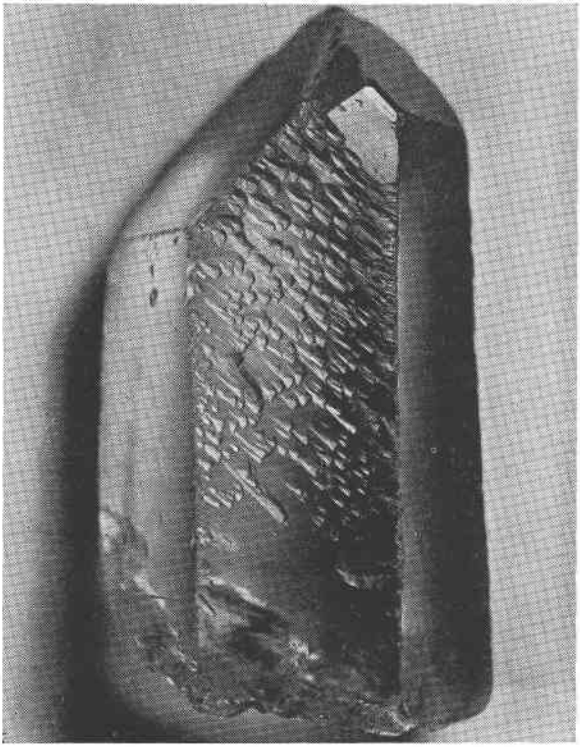


FIG. 8. Natural etch figures on Brazilian quartz. Apices of triangular pits on *m* $(10\bar{1}0)$ point to the major rhomb *r* $(10\bar{1}1)$, the largest apex face in the picture. The deep etch pits on this crystal occur on alternate prism faces, none occurring on the intermediate prism faces. The striations on *s* $(11\bar{2}1)$ are parallel to another *r* face in the upper right hand corner

ORIENTATION BY PARTING

Dana[15] lists as "cleavage: *r*, *z*, difficult and not often observed, also *m*, and sometimes *c*, more difficult; sometimes developed by sudden cooling after being heated; also by the pressure of a sharp point on thin sections, *e.g.*, cut $\parallel c$ and $\perp m$." This parting of quartz on *r* $(10\bar{1}1)$ is skillfully utilized in cobbing quartz in Brazil, and a very practiced eye can set up quartz by this means.

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[15] Dana, VI System, p. 186 (1892).

When a thin Z-section, 0.050″ to 0.100″ thick, is heated a few minutes on a hot plate and slowly immersed in a beaker of water at room temperature, prismatic cracks develop parallel to the X-axes (Fig. 9). The set of parallel, almost equally spaced cracks which first form are those more nearly perpendicular to the water surface. By slowly rotating the section while immersing, sets of cracks parallel to the other X-axes are developed. The cracks grow slowly at a rate dependent upon the rate of immersion, accompanied by a clearly audible cracking sound and their growth may be readily observed. The surfaces are remarkably straight and parallel to the X-axes and often follow a zig-zag path. The section is easily broken along the cracks and the surfaces have a brilliant luster. Rapid immersion and the use of thick sections generally causes a shattering and a multitude of random cracks. The method has been used abroad to determine X in defaced quartz by refitting the cracked section to the crystal.[16]

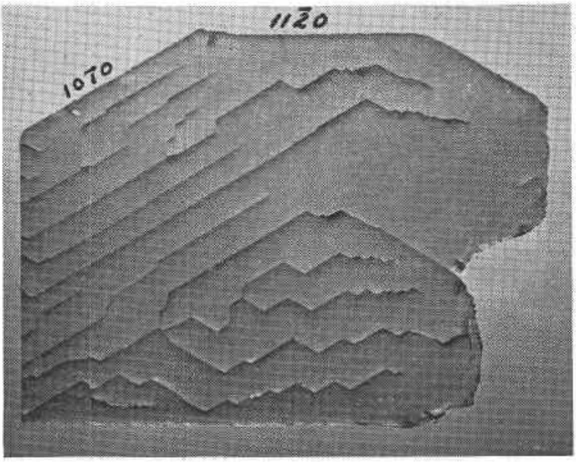

FIG. 9. Prismatic parting parallel to X-axes developed on 0.090″ thick heated Z-section slowly immersed in beaker of cold water.

[16] Booth, C. F., *op. cit.* This type of thermal parting seems to have escaped the attention of mineralogists. See also Judd, J. W., On the development of a lamellar structure in quartz by mechanical means: *Mineral. Mag.*, **8,** 1–8 (1888); Mallard, E., Sur les clivages du quartz: *Bull. Soc. franc. Min.*, **13,** 61–62 (1890); Schubnikow, A. and Zinserling, K., Über die Schlag-und Druckfiguren und über die mechanischen Quarzzwillinge: *Zeit.Krist.* **8,** 243–264 (1932); Drugman, Julien, Prismatic cleavage and steep rhombohedral form in α-quartz: *Mineral. Mag.*, **25,** 259–263 (1939); Fairbairn, H. W., Correlation of quartz deformation with its crystal structure: *Am. Mineral.*, **24,** 351–368 (1939); Frondel, Clifford, Secondary Dauphiné twinning in quartz: *Am. Mineral.*, this issue.

Other methods of less value are no longer used.[17] In one, a small steel ball was dropped upon a basal section from a height of about half a meter. The section was polished and the sides of the dent were approximately parallel to the X-axes. The method has many obvious disadvantages as well as being inaccurate. Rivlin's method[18] consisted of grinding one side of a Z-section with coarse silicon carbide using a rocking and crunching motion. A diffuse hexagonal light figure with sides approximately parallel to the natural prism faces appears when viewed over a pin-hole source of light.

Optical Methods for Orientation of Quartz

The optical methods for quartz crystal orientation comprise the stauroscopic or conoscopic location of the optic axis in faced or defaced quartz, or in sawed material in the process of manufacture.

Mounting Stauroscope.[19] This instrument consists simply of a pair of crossed Polaroids, a source of illumination, and a stage with a reference edge, upon which a quartz crystal, section or bar can be oriented for mounting on a glass plate for sawing (Fig. 10). The approximate direction of the optic axis must be known from morphology of the crystal or by examination in the inspectoscope or conoscope. It is used only in lining up the optic axis of the quartz to about a degree of parallelism with the reference edge of the glass plate. Due to the relatively large size of the quartz masses this is about as accurate an orientation as can be achieved with Polaroid as an analyzer, or the use of supplementary biquartz plates,[20] or through the use of a photoelectric cell.[21] A third piece

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[17] A good example of the complexities introduced in the processing of quartz plates due to the lack of fundamental crystallographic knowledge is seen in some of the elaborate methods developed for determining the electric axes. One method involved cutting a ring perpendicular to the optic axis and applying an alternating electric field in an evacuated chamber and noting the direction of the X-axes from the luminous phenomena (Geibe, Erich and Scheibe, Adolf, Method for determining electric axes in crystals: *U. S. Patent Office* No. 1,720,659, July 16, 1929). Another method involved cutting a Z-section and determining the piezoelectric effect at different parts of the surface by a probing electrode and plotting the results (Dawson, Leo H., Method and apparatus for determining the direction of the electric axes of crystal quartz: *U. S. Patent Office* No. 1,866,454, July 5, 1932).

[18] Rivlin, R. S., Grinding and scratching crystalline surfaces: *Nature*, **146,** 806–807 (1940). The figure is pictured by Willard, G. W., *Bell Syst. Tech. Jour.*, **23,** 36 (1944).

[19] Mounting and angular-view stauroscopes may be obtained from The Polarizing Instrument Co., Inc., 41 E. 42nd St., N.Y.C.

[20] Made from basal sections of right- and left-quartz 4 mm. thick. A piece of transparent Scotch tape may be used as a substitute by cutting a square piece diagonally, turning one half upside down and joining again on the diagonal line. It may be placed on a glass slide below the analyzer or directly on a glass mounted Polaroid. Both halves of the tape will show the same color when the quartz is at extinction, and different colors when not

of Polaroid with marked direction of the plane of polarization may be used to calibrate the reference edge of the stage.

Angular-View Stauroscope. This instrument (Fig. 11) is essential for finding the electric axis direction in wafers to guide in dicing them, and

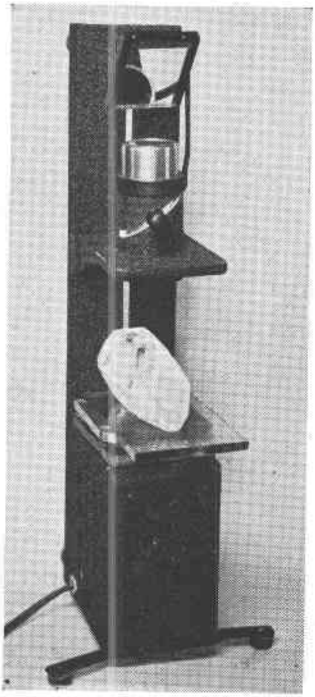

FIG. 10

FIG. 11

FIG. 10. Mounting stauroscope used for setting Z (*c*) of crystal, X-section or bar parallel to long reference edge of glass mounting plate. Instrument is simply a pair of crossed Polaroids set with standards so that plane of polarization of one Polaroid is parallel or perpendicular to reference edge on stage.

FIG. 11. Angular-view stauroscope. Stage set at approximately 45° to tube carrying crossed Polaroids. Only one complete extinction position is obtained for AT- and BT-wafers. The marking guide is used to rule this direction left to right, and an arrow head marked away from the observer to indicate optic axis direction which in this position is approximately perpendicular to tube.

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quite at extinction. Split-field Polaroids with the plane of polarization of each half 3° apart are also available.

[21] Since large amounts of light are available, a photovoltaic type of cell such as the Weston Photronic cell may be used without the necessity of adding an amplifier. See Whitford, A. E., Chap. X, in Strong, John, *Procedures in Experimental Physics*, Prentice-Hall, Inc., New York (**1938**).

for finding the electric and optic axis directions in wafers and blanks preparatory to rechecking them by x-ray measurements. For the latter purpose, the instrument permits setting the wafer or blank in the crystal holder in the proper position for x-ray measurement. It differs from the conventional stauroscope in that the stage is at an angle of about 45° to the axis of the tubes carrying the crossed Polaroids. The field of the polarizer is made small. The instrument is usually equipped with a rule to mark the direction of the electric axis (left to right in the extinction position), and an arrow pointing away from the observer to indicate the optic axis direction.

If a BT-wafer is revolved on the stage it will dim out twice and extinguish completely only in one position; in this position the optic axis direction is perpendicular to the axis of the tube and the electric axis is left to right. When the section is rotated 180° from the extinction position, the line of sight will be parallel to the optic axis as evinced by the characteristic color bands due to rotatory polarization.

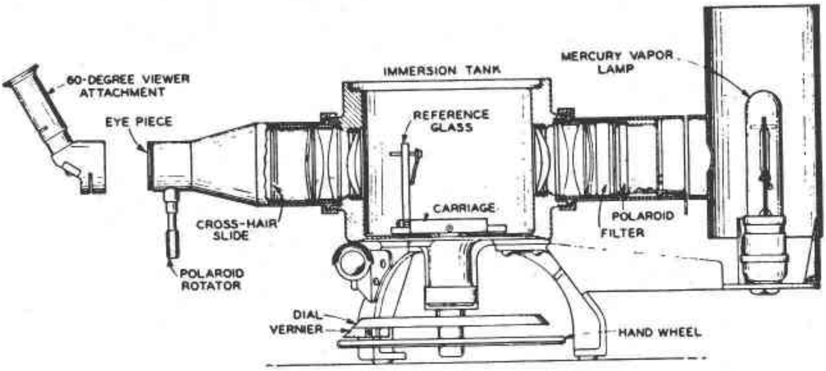

FIG. 12. Conoscope designed at Bell Telephone Laboratories. The instrument is invaluable for rapidly and accurately locating the optic axis of faced or defaced quartz and may also be used to determine hand. (Drawing from W. L. Bond, *Bell Syst. Tech. Jour.* **22,** 224, 1943.)

Conoscope.[22] The conoscope is a device which has been in use by crystallographers for over a century for examining minerals in convergent light and measuring their optic axial angles. For the purpose of the quartz oscillator industry, the instrument has been greatly improved[23] over the crude Fuess model which is occasionally found gathering dust in the older mineralogical laboratories. The optical system has been

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[22] Manufactured by Bulova Watch Co., N.Y.C. and Shuron Optical Co., Geneva, N. Y.

[23] Bond, W. L., *op. cit.*

carefully designed, and the analyzer can be rotated to determine the hand of the quartz (Fig. 12). The trough has a rotating stage on the bottom with a track for a jig or a vertical glass reference plate to carry sections. A graduated circle and vernier permits readings to 6′. The trough is large enough to hold a mass of quartz weighing about one kilogram. The light source is a mercury vapor lamp whose rays are converged by the optical system to enter the quartz at a cone angle of 40°. It is essential that the quartz be immersed in a liquid with an index of refraction of 1.544, and a number of such liquids are listed by Bond. Many of the liquids used have one or more of the objectionable features of being viscous, nasty smelling, skin irritating, or becoming so dark from their dry cleaning action on the quartz and air as to become opaque. Lindol[24] (ortho-free tricresyl phosphate) has an index of 1.555 (at 20°C) and may be lowered to 1.544 by mixing with a miscible liquid of lower index; it seems to have the only fault of being viscous.

Quartz crystals, faced or defaced, and sections may be quickly oriented by centering the optic axis figure upon the cross-hairs (one horizontal and three vertical) in the eyepiece. The image is an interference figure due to axial birefringence[25] resulting in rotation of the plane of polarization. Rays differing in convergence pass through different thicknesses of quartz and each colored ring represents those rays which emerge at the surface of the quartz vibrating parallel to the plane of polarization of the analyzer. The color of the center of the first circle, is the complement of that absorbed by the interference of the two circularly polarized rays travelling in the direction of the optic axis. The color of the rings is due to interference of elliptically polarized rays; only at the farthest rings where isogyres appear are the effects of interference of plane polarized rays, ϵ and ω to be seen. Although optical twinning confuses the pattern, the rings remain clear enough for orientation.

Z-Sections in the Conoscope. The first use that seems to have been made of the conoscope was the testing of the trueness of a plane cut perpendicular to the optic axis. The stage was set a' 0° with a vertical glass plate in the tank normal to the beam of light. The section to be tested was held against the glass reference plate and the position of the optic figure was observed. If the saw cut was true, the rings would center about the cross-hairs (Fig. 13). Any divergence would be apparent and could be measured by rotating the Z-section upon the glass plate with the stage set at 0°, until the center of the rings had travelled to the maximum distance from the cross-hairs, and then reading the circle. Since two planes intersect

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[24] Sold by Celluloid Corp., 180 Madison Ave., N.Y.C. Specify ortho-free for the orthoform is toxic.

[25] On the axial birefringence of quartz, see Sosman, Robert B., *op. cit.*, pp. 667–669.

in a straight line, it is obvious that in the position of maximum divergence of Z, the intersection of the true (0001) plane (the desired cut) and the plane which has been cut (resting against the glass reference plate) is now vertical, and that this vertical line is one of zero deviation. The maximum divergence is at right angles to this line and in the direction of the optic figure seen in the conoscope. This direction is marked on the section, which can be taken to a lapping wheel and lapped on the "high side"; jigs have been developed for the purpose. The section is rechecked until no divergence is apparent.

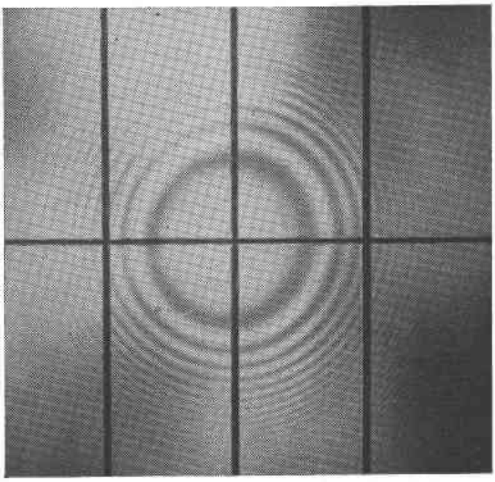

FIG. 13. Series of rings centered on cross-hairs of conscope show optic axis is parallel to reference track in immersion tank.

Mounting Defaced Quartz in the Conoscope. Because the optic axis direction may be so readily found in the conoscope, and so accurately centered, the instrument has been invaluable in cutting defaced quartz. It permits cutting at least one true plane, perpendicular to the optic axis, which is easily accomplished, after which the quartz can be cut as faced quartz.

For mounting purposes, a ground surface of about one square inch parallel to the optic axis is desirable. The quartz is turned in the oil in the conoscope until the optic axis is centered; is carefully lifted out, and without losing the optic axis direction, is pressed down on a large iron lap carrying wet #100 silicon carbide. The surface is wiped clean, and the quartz is set in the bottom of the conoscope to make sure that the mounting surface is not more than about 10° from parallelism with Z. The optic axis direction is marked by a line drawn on the ground surface cemented to a glass plate with Z approximately parallel to the reference edge using the mounting stauroscope. The mount is locked to a simple transfer jig, which fits on the track in the conoscope. Screw adjustments on the jig permit tilting the mount until the optic axis figure is on the horizontal cross-hair. Divergence from the vertical cross-hair may be read on the graduated circle and marked on the plate for correction at the saw.

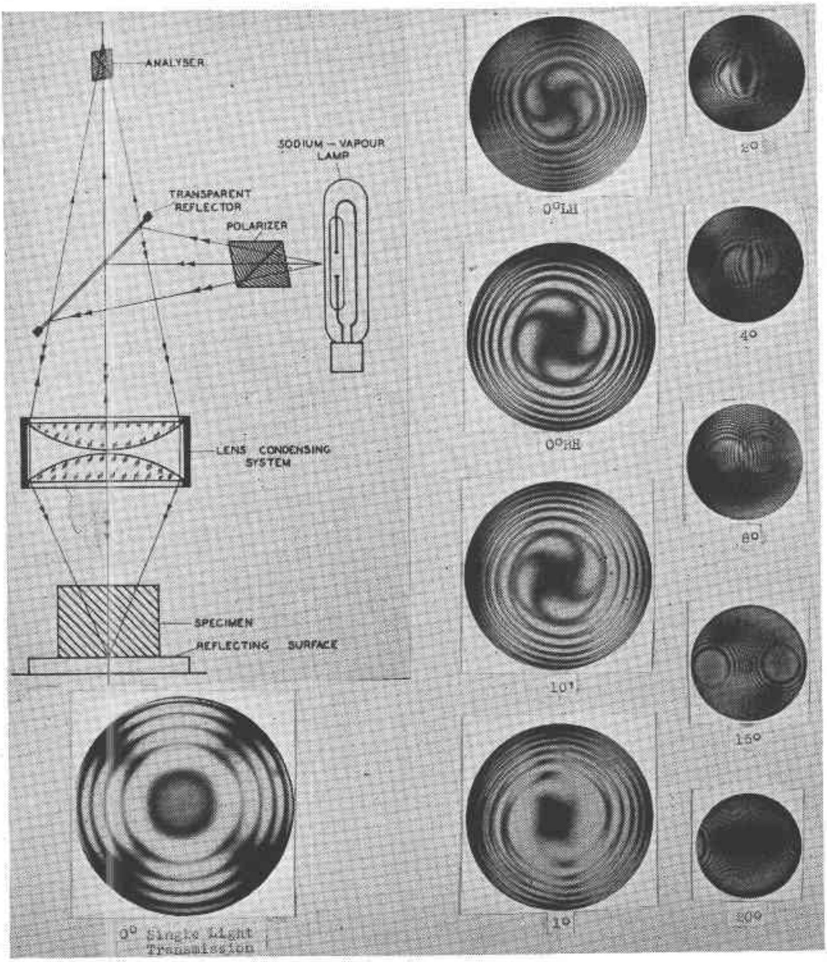

FIG. 14. Airy-spiral conoscope. A novel instrument developed in England for testing trueness of Z-sections. On single light transmission the figure (lower left) is similar to that seen in conoscope. On passing light back through the section by means of a mirror, the Airy-spiral figure is observed. The latter is sensitive to slight inclinations of the optic axis as indicated by the series of figures for various inclinations. (From C. F. Booth, *Jour. Inst. Elect. Eng.*, **88,** 97, 1941.)

Airy-Spiral Conoscope. This conoscope, of novel design, was developed in England for testing the trueness of a Z-section.[26] The Z-section is placed upon a mirror which can be revolved (Fig. 14). Light from a monochromatic source such as a sodium vapor lamp is polarized, reflected by a thin glass plate through condensing lenses to the Z-section. The mirror reflects the light back through the quartz to the analyzer and the image seen is like that of the well known Airy's spirals formed by superimposing a basal section of right-quartz upon a basal section of left-quartz in convergent polarized light. The figure is very sensitive to errors of angle in cutting the Z-section which are detected by the divergence of the two images.

Projection Conoscope. If a Z-section of quartz, a few mm. thick is sandwiched between a pair of crossed Polaroid films, and held close to the eye, the optic axis figure due to rotation of the plane of polarization can be seen. By holding a point source of light (such as a projection or automobile light bulb) close to the sandwich, it is possible to project the image to a not too distant screen. It is not difficult to rig up a device to project the optic axis image of Z-sections upon a screen which can be ruled to indicate divergence from trueness.

ORIENTATION OF ETCHED QUARTZ

The etching processes are extremely important in the manufacture of quartz oscillator-plates. The pin-hole light figures are used for *orientation* of blocks, bars and sections. Raw crystals, blocks, and wafers are etched to reveal *twinning*.[27] In the *final finishing* operations, the blanks are etched to frequency; this is an ideal finishing procedure[28] since it results in a stable surface which does not "age."

Etching Conditions. Hydrofluoric acid was used almost exclusively in the quartz industry up to a few years ago but its use caused serious industrial hazards and inconveniences. Today nearly all manufacturers use a concentrated water solution of ammonium bifluoride. The elaborate precautions required in the use of HF are not necessary in using the bifluoride although certain safety measures should be followed in using all fluoride reagents. At ordinary room temperatures, 33 gms. in 100 cc. of water is practically the limit of solubility. The reaction is strongly endothermic and solution is promoted by warming the water. HF etches quartz two to three times faster than ammonium bifluoride. Although their pin-hole light figures differ in some details the general description given here applies to figures obtained with both solvents. In general HF produces larger and coarser etch pits and the bifluoride forms a larger number of finer etch pits. Many other solvents have been used to etch quartz such as potassium carbonate, sodium fluoride, etc.

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[26] Booth, C. F., *op. cit.*

[27] Gordon, Samuel G., The inspection and grading of quartz: *Am. Mineral.*, this issue.

[28] Frondel, Clifford, Etching techniques in the manufacture of quartz oscillator plates: *The Radio Eng. Digest:* **1**, 31–36 (1944); Final frequency adjustment of quartz oscillator-plates: *Am. Mineral.*, this issue.

Natural crystal faces are very slowly etched but roughing the surfaces by lapping or sand blasting prior to etching decreases etching time and produces more evenly distributed and better developed etch figures. The coarser the abrasive used the shorter is the etching time. In etching-to-frequency in the final finishing operation where only 0.00004″ to 0.00005″ is removed for final frequency adjustment and to effectively prevent ageing, the machine lapping finish must be carefully controlled to obtain predictable and reproducible results. Crystals should be cleaned in a solvent such as carbon tetrachloride or trichlorethylene ("Tromex") before etching. When holes, crevices and blue needles are present, the quartz is rapidly etched to a fibrous aggregate.

The etching rate increases markedly with temperature. Oscillator plates with #303 optical flour machine lap finish decreased approximately 0.00056″ in thickness when etched two hours in a 30% solution of ammonium bifluoride at 75°C. compared to approximately 0.00014″ being etched off under the same conditions at 25°C.[29] Large masses of quartz such as raw crystals, *X*-blocks, *Z*-sections, etc., must be etched at or near room temperatures to prevent cracking due to differential thermal expansion. At least ten to twenty hours etch is required to produce a surface for orientation and inspection purposes. Rough-cut wafers obtain an etch sufficient for twinning inspection in 1½ hours at 50°–60°C. in a 30% ammonium bifluoride solution. Agitation of the etch solution by rocking the container or constant stirring also speeds the action. The thickness decrease of crystals etched under the conditions stated above at 75°C. but with the etch solution constantly stirred is approximately 0.00071″.

The tendency in the crystal industry has been to stop etching as soon as the matt surface appears. The first effect of etching is to dissolve the thin submicroscopic surface layer which has been shattered by sawing or lapping and better results are obtained if the etching is continued into the solid quartz.

Etch Figures on Quartz. Solution is much more rapid (by a factor of over 100 times) parallel than perpendicular to the optic axis.[30] The minor rhombohedral (*z*) faces are more rapidly etched than the major rhombohedral (*r*) faces; the latter give a coarser and more geometrical pattern (Fig. 19). Kalb[31] and Ichikawa[32] have studied natural etch figures. There have been several classical studies on the etching of spheres,[33] crystals and sections.[34]

[29] These thicknesses were computed from frequency measurements using the formula: Thickness (inches) = K/Frequency (kilocycles) where $K = 100$. ½″ square BT-cut crystals, 7.8 MC starting frequency.

[30] Mügge, O., *Rosenbusch Festschrift*, 96–126, Stuttgart (1906).

[31] Kalb, George, Beitrage zur Kristallmorphologie des Quarzes. I–V: *Zeit. f. Krist.*, **86,** 439–452 (1933); **86,** 453–465 (1933); **90,** 163–185 (1935); also **86,** 1–7 (1933); **89,** 400–409 (1934); also literature references contained therein.

[32] Ichikawa, S., Studies on the etched figures of Japanese quartz: *Am. Jour. Sci.*, **39,** 455–473 (1915).

[33] Meyer, O., and Penfield, S. L., Results obtained by etching a sphere and crystals of quartz with hydrofluoric acid: *Trans. Conn. Acad.*, **8,** 158–165 (1889). (Note remarks of Van Dyke, p. 403 of article cited below, regarding hand of crystal used.) Gill, A. C., Beiträge zur Kenntniss des Quarzes: *Zeit. Kryst.*, **22,** 97–128 (1893). Mugge, O., *op. cit.* Nacken, R., Atzversuche an Kugeln aus Quarz und α-Quarz: *Neues Jahrb. Mineral.*, **1,** 71–82 (1916). (Also describes etching of spheres in sodium metaphosphate at 700°C. to determine symmetry of high temperature quartz). Bond, W. L., Etch figures of quartz: *Zeit.*

Photomicrographs of typical etch figures on various planes of quartz and their orientation are shown in Fig. 15. Sections (*a*) to (*e*) show etch figures on both sides of an electrical twin boundary indicated by pairs of lines on the rim of the photograph. These sections were cut 0.095″ thick and lapped successively in #320 and #600 SiC and finally 0.002″ lapped off with #303 Al_2O_3. The sections were thoroughly cleaned and etched 4 hours with 30% NH_4HF_2 at 50°C. Section (*f*) is an untwinned section lapped only with #600 SiC and etched 2 hours under the same conditions. (*a*), (*b*) and (*c*) were cut at 35°15′ (AT), 38°13′ (major-minor rhomb plane) and 49°30′ (BT), respectively, to Z which is indicated by the arrow and is inclined to the plane of the paper. The characteristic "shingle" structure is on the side more nearly parallel to the major rhomb *r*, the "ripple" structure on the side nearly parallel to the minor rhomb *z*. The characteristic parallelogram light figure described below is seen as being due to parallelogram-like etch figures (*d*) on that side of the X-plane with −X up. The indistinct types of etch figures on the +X side of the X-plane produce the arrow-like light figures. The Y-plane shows etch figures (*e*) pointing in opposite directions along Z on either side of the electrical twin boundary. The characteristic triangular light-figure patterns on etched Z-sections is seen to be due to distinct and geometrical etch figures which appear like sections of rhombohedra standing on a corner forming a triangular figure in horizontal sections. The edges between the rhomb faces appear to be parallel to the Y-axes. In an electrical twin these figures are rotated 180° about Z.

Orientation of Quartz by Etching. The characteristic symmetry expressed in well developed quartz crystals by the disposition of the zone of minor faces *x* $(51\bar{6}1)$, *s* $(11\bar{2}1)$ and *z* $(01\bar{1}1)$ above the negative (on compression) end of the polar X-axes is shown also by the symmetry of the etch figures on natural crystals and those produced by solvents on sections of quartz. Diagnostic also are the pits etched on the *r* and *z* faces and since these planes become coplanar in twinned crystals it is a

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Krist., **99,** 488–498 (1938). (Gives photomicrographs of etch figures on a hollow quartz sphere etched with HF.) Van Dyke, Karl S., On the right- and left-handedness of quartz and its relation to elastic and other properties: *Proc. Inst. Radio Eng.*, **28,** 399–406 (1940).

[34] Descloizeaux, A., Memoire sur la cristallisation et la structure interieure du quartz: *Ann. Chim. et Phys.* (3) **45,** 129–316 (1855); *Memoires Acad. Sci.*, (Inst. Imp. France) **XV,** 404–614 (1858). Leydolt, Franz, Über eine neue Methode, die Struktur und Zusammensetzung der Krystalle zu untersuchen, mit besonderer Berücksichtigung der Varietaten des rhomboedrischen Quarzes: *Sitzber. d. kais. Akad. d. Wiss. Wien* (*Math. Natur. Classe*) **XV (1),** 59–81 (1855). Molengraaff, G. A. F., Studiem über Quarz: *Zeit. Kryst.*, **14,** 173–201, 1888; **17,** 137–176 (1889). Bomer, A., Beitrage zur Kenntniss des Quarzes: *Neues Jahrb. Mineral. B. B.*, **7,** 516–555 (1891). Honess, Arthur P., *The Nature, Origin and Interpretation of the Etch Figures on Crystals*, John Wiley & Sons, New York (1927). Booth, C. F., *op. cit.*

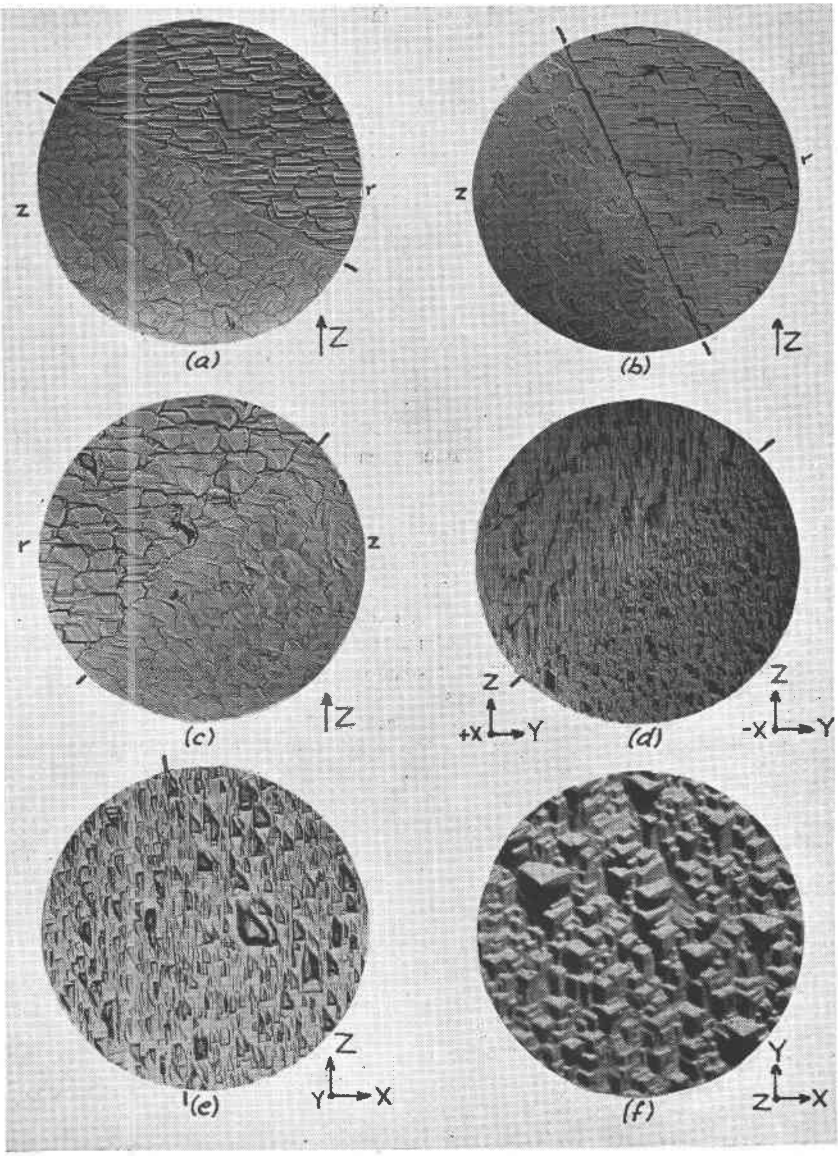

FIG. 15. Photomicrographs of etch figures on quartz produced on finely lapped surfaces etched with NH_4HF_2 (see text). Electrical twin boundaries in (a) to (e) indicated by pairs of lines on periphery of each photograph. (a) AT-plane, ZZ′=35°15′, magnification 60×; (b) Major-minor rhomb plane, ZZ′=38°13′, 60×; (c) BT-plane, ZZ′=49°30′, 60×; (d) X-plane, 110×; (e) Y-plane, 110×, (f) Z-plane, 175×.

method of distinguishing them. The etch pits, or their refracted and reflected light pattern figures, are the sole infallible and unambiguous criterion for the orientation of quartz.

The Abbe C. Gaudefroy[35] in 1931 called attention to the photographs of DeGramont and Mabboux[36] and the use of light figures seen on reflecting a distant light source (several meters away) from HF etched basal sections of quartz for fixing the position of the 2-fold and 3-fold axes to about one degree, the sense of the piezoelectric axes, and the hand of the crystal even in defaced quartz. In 1933, Gaudefroy described in detail the now familiar parallelogram and arrowlike light figures which he called *motif asterique*, and which he obtained by reflection from etched spheres of quartz.[37]

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[35] *Bull. soc. franc. min.*, **54,** 80 and 108 (1931).

[36] "Supplement a la notice sur les travaux scientifiques de M. Armond de Gramont, *cit.* Paris, 1929."

[37] Gaudefroy, C., Sur les groupements de cristaux de quartz à axes parallèles: *Bull. soc. franc. min.*, **56,** 5–63 (1933). Light figures of etched spheres of quartz were used by Nacken, *op. cit.*, for showing the symmetry of quartz.

The posthumous work of Armond De Gramont, *Recherches sur le quartz piezoelectrique*, Ed. de la Rev. d'Optique Theorique et Exp., Paris (1935), seems to have been the inspiration for some of the orientation devices in use. Several devices and methods have been recently described and some have been assigned patents for use in orienting etched sections and crystals by means of their light figures:

Hawk, Henry W. N., Art of examining quartz: *U. S. Patent Off.* No. 2,264,380, Dec. 2, 1941. One side of a *Z*-section is etched and the other remains unetched or is polished. Unlike most of the other methods of using the light figures, the etched side is toward a pin-hole source of light and the figure is observed from the unetched side of the image or is projected on a screen. The method claims to determine the *X*-axes which are parallel to the sides of the observed equilateral triangle, their polarity, and direction of the major rhombohedral faces from the position of the triangle.

Anon., *Manual for the construction and use of the rodometer*, Scott Lab., Wesleyan Univ., Middletown, Conn., April 1943. This paper describes more elaborate equipment in which the light figure is projected on a ruled screen by means of a photographic lens. The unetched end of the crystal is immersed in oil to reduce refraction and reflection. The great difficulty in applying such methods to actual production is that in order to transfer the orientation to a saw, it is necessary to employ gimbals and the like to hold the crystal for cementing and processing. See for example Waesche, Hugh H. and Wolfskill, John M., A method of orientation and sawing small unfaced quartz: Inf. Bull. No. 5, *Handbook for the Manufacture of Quartz Oscillator-Plates*, Off. Chief Sig. Off., War Dept., Washington, D. C., March 1943.

Several devices are described by Willard, G. W., Use of the etch technique for determining orientation and twinning in quartz crystals: *Bell Syst. Tech. Jour.*, **23,** 11–51 (1944). These include a *reflection oriascope* (from orientation- and scope) for detecting the sense and approximate direction of the electric axes. The rays of light are brought to focus by a pair of condensing lenses on the surface of an etched *Z*-section and reflected back upon a viewing screen. The trigonal figure is shifted about 12° clockwise or counterclockwise from the *X*-axes depending upon the hand of the crystal and ruled with a preset marking

Production of Light Figures on Etched Sections. The characteristic light figures are the result of reflecting and refracting light rays from the facets of the etch pits or hills. The figure obtained is that of the upper etched surface of the section. If the lower surface in contact with the pin-hole disturbs the effect, it may be immersed in a layer of oil in a glass vessel placed upon the pin-hole. The image is a composite of several images and is not in one plane. In photographing it, it is necessary to focus on a plane about half way in the section. A point source of light is used such as a distant light, an illuminated pin-hole, or a light brought to a focal point by a condensing lens. One of the best methods of observing the figure is to place the section on the stage of an ordinary microscope, remove the entire microscope tube and illuminate the section by means of the condenser (Fig. 16). The most commonly used device is a box containing an electric light bulb which illuminates a tiny pin-hole (made with a #60 or smaller drill) in a sheet of metal. Sometimes several pin-holes are used to obtain greater accuracy in ruling the axial directions; each pin-hole produces a separate figure. A hinged ruler is convenient for marking directions of the axes.

Light Figures on Etched Z-Sections. The figures obtained upon etching basal sections of quartz vary markedly with the preparation of the surface and etching time. The figures shown in Fig. 17 were obtained on a

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template. In the *pin-hole oriascope,* light is passed through a pair of condensing lenses to a mirror which reflects it to a diffusing screen in contact with the pin-hole. Preset marking guides are provided for ruling axial directions on Z- or X-sections.

In the *reflection twinoriascope,* used for detecting usable portions of electrically twinned AT, BT, CT and DT wafers, the mounting table is tilted to an angle depending upon the angle of cut of the wafer being examined. Deeply etched wafers also show a characteristic light reflection figure when examined in a beam of light brought to focus by a condensing lens. These figures distinguish parts of electrically twinned wafers as being cut respectively at negative or positive angles to Z: those on the negative angle side have the appearance of a golf club (usable side in case of BT- and DT-wafers); the positive angle side shows the silhouette of a spoon (usable side for AT- and CT-wafers).

Gerber, P. D., Piezoelectric crystal: *U. S. Patent Off.*, No. 2,218,489, Oct. 15, 1940; Apparatus for examining quartz: *ibid.*, No. 2,313,143, March 9, 1943. The first patent describes a method for locating and determining the polarity of X-axes and the hand (the Herschel convention is used) of etched Z-sections and the second patent describes a device for this purpose. The etched Z-section is placed on a rotating stage and light from a projector at 30° to the surface is observed at an angle of 10° to 15° from the horizontal surface. It is stated that the sharpest reflections for this set-up are obtained 10° to 15° clockwise or counterclockwise from $-X$, depending upon the hand of the crystal (note similarity to the above description). A ruling guide is provided for marking the specimen.

Eckert, J. F., Art of examining quartz: *U. S. Patent Off.* No. 2,328,968, Sept. 7, 1943. This patent relates to the use of a parallelogram light figure observed on a pin-hole light box but contains a serious error. The long side of the parallelogram is stated to be the direction of Z, and the short side *r*. These, of course, are actually just in reverse.

surface cut with a 12″ Norton metal bonded diamond blade (but not lapped) and etched 28 hours in 30% ammonium bifluoride solution at 68°F. Z-sections prepared in this way show two characteristic sets of three spots. By drawing connecting lines between the spots of either set,

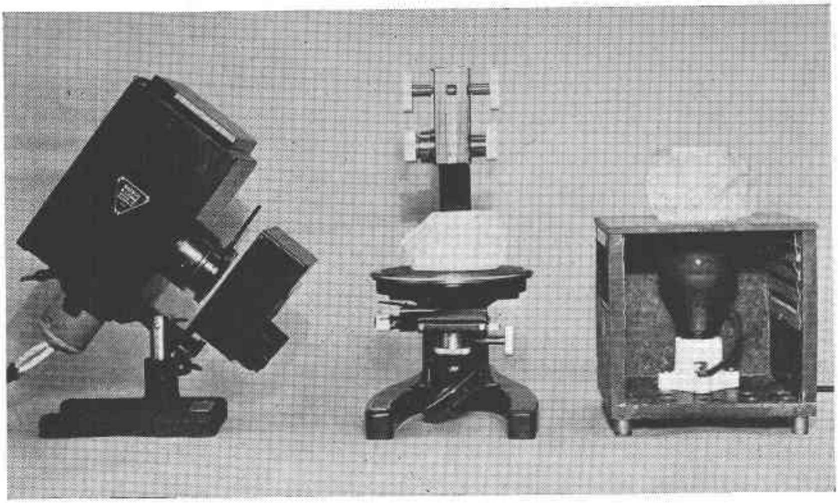

FIG. 16. Light figures of etched sections may be observed by placing section on an illuminated pin-hole. A microscope stage with tube removed and condenser in place (left) or a box with a 60-W light bulb and a #60 drill hole (right) may be used.

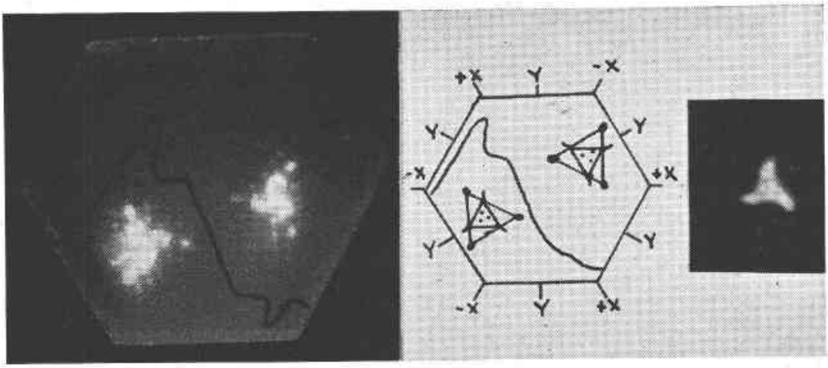

FIG. 17. Light figures on electrically twinned Z-sections showing three typical equilateral triangles formed by three sets of spots useful for orientation purposes. The outer set of brightest spots form equilateral triangle with sides parallel to Y-axes. The fainter spots with tails form a triangle with sides parallel to X-axes. The propeller-like figure becomes more distinct with long etching and shows hand of crystal (both right-handed in this case).

they form a perfect equilateral triangle when the section is perpendicular to the optic axis. A faint group of three spots with tails are at the corners of a small equilateral triangle whose sides are parallel to the X-axes. The tails of the spots are in the direction of the Y-axes. A larger equilateral triangle is formed by three very much brighter spots and its sides are parallel to the Y-axes. In electrical twinning the small triangles are related as by a mirror image and the large triangles are reversed 180°. Other details appear as shown in the photograph but these have not yet been analyzed.

With a longer etch, both sets of dots practically disappear and the trigonal trapezohedral symmetry becomes more obvious. The figures assume the shape of three-leaved propellers which show the hand of the crystal (right-hand screws for right quartz, etc.), the arms roughly indicating the direction −X. These propellers are also visible but less apparent in sections with a shorter etch and are most distinct on sections which have been lapped prior to long etching. When prepared in the latter way, a third set of dots become apparent in the center of the propeller (which becomes more distinct) and forms a tiny equilateral triangle. The sides of this triangle are rotated approximately 13° ± 2° from the Y-axes. These two triangles are related by a 180° revolution in electrical twinning.

The orientation of the light figures is used to determine the approximate direction of Y, which is ruled on the section. An accurate determination may be made by *x*-ray reflection from the side of the Z-section using the $(11\bar{2}0)$ or $(10\bar{1}0)$ atomic planes and the right angle marking guide as described in the following section.

The direction of the electrical axes is precisely indicated by the twin boundaries of optical twinning if such twinning is present. Electrical twinning is identifiable as irregularly bounded areas.

Orientation of Etched X-Sections and Y-Bars. The "parallelogram" light figure obtained on viewing the negative (on compression) side of an etched X-section laid on an illuminated pin-hole, has proven to be a most useful aid in the precise orientation of quartz (Fig. 18). It indicates the trend of a zone of minor faces *x* $(51\bar{6}1)$, *s* $(11\bar{2}1)$ and *z* $(01\bar{1}1)$, were these on the crystal. The long, fainter sides of the parallelogram mark the slope of the major rhomb faces; i.e., they are parallel to the intersection edge of *r* $(10\bar{1}1)$ and the plane of the section $(11\bar{2}0)$. The shorter brighter sides are parallel to the optic axis direction, (Z, *c*). The positive side of the X-section shows a figure often referred to as the "*Z*" or arrow-figure. The horizontal line indicates the Y-axis and the two appendages at either end show the direction of the minor rhomb faces.

Since AT, CT, ET and GT cuts are not far from parallelism with the minor rhomb face *z*, Fig. 1, while the other standard cuts, BT, DT and

FT are not far from parallelism with the major rhomb, *r*, and since the trace of the rhomb plane as well as the optic axis direction are so clearly defined by the parallelogram light figure, an unambiguous and infallible guide is furnished for marking the directions of the cuts, without the bother of determining the hand of the quartz in a polariscope or the polarity of an electric axis in the piezometer. A simple rule, useful in directing the cutting of X-sections is given in the procedure of the accompanying paper by the writers on Cutting Schemes.

At the same time, examination of the etched X-section will reveal electrical twinned areas, which may be sawed apart, if of sufficient size to yield blanks since electrical twins are composed of reversed individuals, and therefore one portion of a wafer cut across both would be a miscut on the wrong side of the optic axis. Once the crystal has been cut at an angle to the optic axis, electrically twinned sections cannot be salvaged.

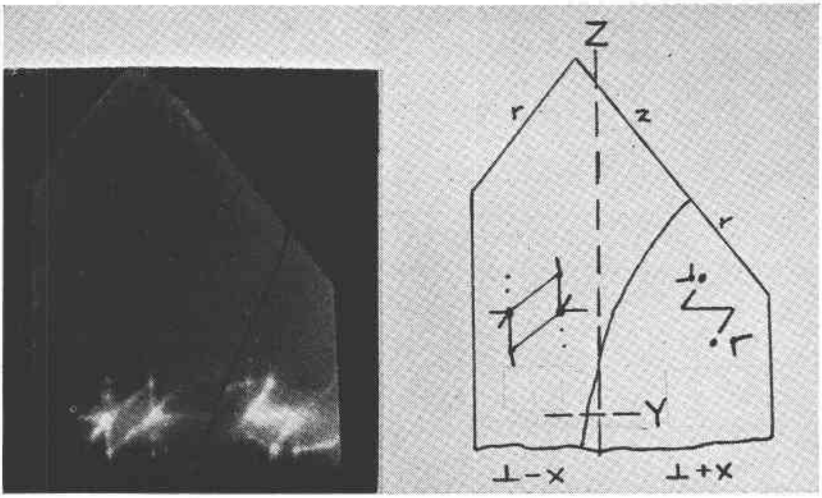

FIG. 18. Light figures on electrically twinned X-section showing characteristic parallelogram light figure when viewed along −X and "*Z*" figure viewed along +X. The use of these figures eliminates the necessity for hand or polarity determinations and are an infallible guide for orientation purposes.

Orientation of Etched Wafers. Wafers are etched for two purposes: to identify and permit the marking out of optical twinned areas and to identify the useful areas of electrical twins. In electrical (180°) twinning, the *r* and *z* faces are coplanar so that one side of a twin boundary on a wafer is usable, while the other side will be equivalent to a miscut of twice the angle of the cut. Pertinent data taken from Willard for correct and miscuts are given in Table 1.

TABLE 1[a]

Cut	ZZ′	Frequency Constant (Freq. (KC)× Thick. (mm.))	Temperature Coefficient (Parts/10^6/C.°)
AT	+35°	1670	0
	−35°	2400	+30
CT	+38°	3080	0
	−38°	2100	−30
BT	−49°	2560	0
	+49°	1880	−55
DT	−52°	2060	0
	+52°	2850	+45

[a] Data from G. W. Willard, *op. cit.*, p. 48.

As was pointed out above, AT- and CT-cuts are near parallelism with *z* while BT-cuts are near *r*. Since *r* and *z* develop different patterns when they are etched, it is a simple matter to distinguish the two planes by

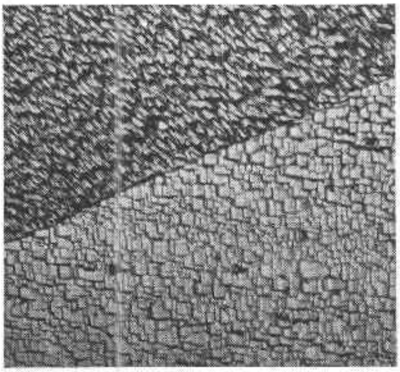

FIG. 19

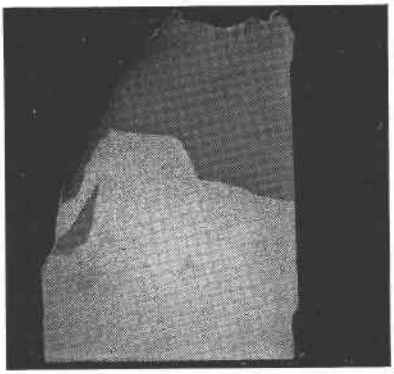

FIG. 20

FIG. 19. The useful portion of a well-etched electrically twinned wafer may be determined by its translucency. The useful side for BT-cut wafers, below, is nearly parallel to *r* has the "shingle" structure, is more translucent than that parallel to *z* which has the "ripple" structure and is the useful side on AT-cut wafers. This wafer was etched very deeply in HF, one surface polished, placed in a slide projector and the image focussed on a screen which was photographed.

FIG. 20. The difference of reflectance from well-etched electrically twinned wafers is applied in determining the useful portion. When examined in a twinoscope the useful side of BT-cut wafers reflects brilliantly once in a 360° revolution when viewed at about 22° from the incident light beam.

their etch figures, either in misoriented crystals, or in coplanar electrical twins.

If a well-etched electrically twinned wafer is examined under a binocular microscope, or Scotch-taped to a glass slide and projected on a screen, the characteristic patterns of the two planes will be apparent. Etched major rhomb faces ($r\ 10\bar{1}1$) show striking rhombohedral facets, which may be described as a *shingle* structure (Fig. 19); the minor rhomb faces ($z\ 01\bar{1}1$) have a less definable pattern which may be designated a *ripple* structure. The AT- and CT-types of plates must be diced from areas showing the ripple structure, while BT-plates must be diced from areas showing a shingle structure, provided, of course, that the wafers have been *cut at the proper angle.* If the latter condition has been followed, only one side of the twin boundary is usable, and the other side is worthless.

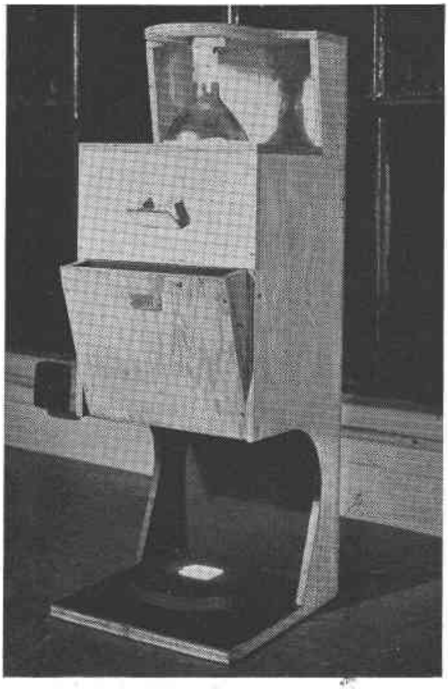

FIG. 21. Twinoscope. Consists simply of a spot-light, turntable and viewing slot approximately 22° from the normal for determining useful portions of electrically twinned wafers by differences in light reflectance.

In actual practice, wafers are examined in a spot-light set at an effective angle, or a *twinoscope* (Fig. 21) which merely consists of a vertical case containing a light which can be adjusted to strike a wafer on a stage (which can be revolved and is painted black) at an angle to cause reflection from the etch figures of the wafer. The useful side of AT-wafers has a frosty, more opaque appearance and the useless side is more translucent and has a brilliant, almost metallic luster. When BT-wafers are revolved on a stage in the spot-light beam, the useful side will reflect strongly *once* (Fig. 20) in a 360° revolution, provided the position of the spot-light has been properly preset by means of a standard. The reflecting r $(10\bar{1}1)$ planes produced on etching are at about 11° to the normal of a BT wafer. The usual routine is to roughly pencil the twin boundary, and to scribble over the flawed areas, including any parts showing optical twinning.