CHAPTER V

Use of the Etch Technique for Determining Orientation and Twinning in Quartz Crystals

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5.1 INTRODUCTION

THE manufacture of piezoelectric plates from crystalline material involves orientation problems not encountered in the fabrication of objects from non-crystalline materials. The reason for this is that crystalline materials have physical properties which vary with the orientation, or direction, in which they are measured.[1] Since the operating characteristics (activity, frequency, and temperature-coefficient) of the finished piezoelectric plate depend, not only upon the shape and dimensions of the plate, but upon the physical properties (electrical, elastic and thermal) of the crystalline material, the finished piezoelectric plate must have a *specific orientation* with respect to the material as well as a specific shape and dimensions. In the case of quartz piezoelectric plates the orientation problem is complicated by two factors. First, a large portion of the available natural quartz crystals lack such natural faces as are required to determine accurately the structure-orientation from the shape of the original stone. Thus the raw stones must be examined for structure orientation by physical instruments before even the first cuts may be made. Secondly, a large portion of natural quartz crystals are twinned, i.e. not of the same structure orientation throughout the stone. The boundaries of the respective, homogeneous regions are not predictable, and cannot be completely located in the uncut stone. Thus the processing of quartz involves a step by step examination for twinning boundaries and orientation as the raw stone is cut into sections, the sections cut into bars or slabs, and the bars or slabs cut into blanks. Even when using untwinned stones the orientation must be redetermined and corrected at each cutting step when making such plate types as require very exact orientation.

The most widely used methods of determining the structure orientation of quartz are: (1) by optical effects (birefringence and rotatory power), (2) by X-ray reflections from atomic planes, and (3) by the use of etch pits which

[1] In crystalline quartz, for example, the electrical conductivity varies with the direction of measurement over a range of 200 to one. The ranges for some other properties are: thermal conductivity 2 to one; linear thermal expansion 1.75 to one; Young's modulus 1.60 to one; piezoelectric constants one to zero; dielectric constant 1.01 to one; index of refraction 1.006 to one; optical rotatory power one to zero.

are developed when the quartz surface is etched in fluorine compounds. Other methods are or may be used in rather special cases. For example, in finished plates of known orientation types, the electrical axis direction is distinguished from other directions by electrical polarity tests (on tension or compression), or a plate known to be one of several types may be tested in an electric circuit for activity, frequency and temperature-coefficient, to determine which type it is. The selective fracture characteristics of quartz offer another method of determining orientation. Microscopic fractures resulting from grinding a quartz surface may be used for determining orientation. Thus unetched, ground, Z-cut surfaces of quartz give a hexagonal figure, when examined by pinhole illumination, which may be used to determine the approximate orientation (but not sense) of the electric axes.[2]

By optical methods (see Chapter II) it is possible to determine the orientation of a quartz body relative to only one direction of the structure, the optic or Z axis. Thus optical methods are limited to determining the angle between the optic axis and a line or surface of the body (but not the rotation of that line or surface about the optic axis). Twinning of the "optical" variety may be detected optically, even when located internally, but the determination of its location in depth is approximate.

By X-ray methods (see Chapter III) it is possible to determine the structure orientation of a quartz body exactly and completely. However, this method is limited in application by the complexity of analysis, except when the approximate orientation is already known. Though twinning can be detected on the surface of the body, it is not generally feasible to explore the surface to locate twinning boundaries. Further, though positive or negative sense of angular orientation is obtainable by X-rays, this part of the complete determination is not reliable unless the specimen examined is known to be free of twinning, or unless the twinning boundary locations are known. Thus X-ray determinations of orientation are generally limited to determining exact orientations in quartz bodies of approximately known orientation (which includes the case in which only one axis is approximately known).

The etch method of determining orientation is commonly used in conjunction with the optical and X-ray methods to give the information that those methods do not give. The etch method, as most commonly and practically applied, does not give exact orientation angles, nor is it applied to specimens of entirely unknown orientation. However, when a surface of approximately known orientation is etched, it is possible to determine approximately the complete orientation (including sense) of the specimen, and further to detect at this surface both electrical and optical twinning and to determine exactly the twinning boundary locations. The detection of twin-

[2] See Fig. 5.20, and further explanation at the end of Sec. 5.53.

ning and twinning boundaries by this method has been practiced for years. The determination of orientation and sense of orientation has been exploited only more recently. At present the etch methods play an important and extensive role in the processing of quartz plates, not only in the routine determination of orientation, but also in the detection of twinning so that the most economical cutting methods may be practiced.[3]

5.2 Twinning (General)

Although the problems related to twinning are largely those of determining orientation of the crystal structure, the nature and prevalence of twinning in crystal quartz presents a special group of problems that would be absent were the twinning absent, and hence are separately grouped as twinning problems. As pointed out in Chapter IV, there are only two common types of twinning in the commercial quartz used for piezoelectric plates, namely, electrical and optical twinning. A simplifying feature of both these types is that the structure axes (optic axis and electric axes) of all portions of a single crystal are parallel each to each. However, they are not of the same sense, or handedness. The difference between the two types is as follows:

In a crystal which is only ELECTRICALLY TWINNED, the crystal is entirely of one handedness (either right or left), but one portion is of OPPOSITE ELECTRICAL SENSE to another portion, i.e., the electric axes are of opposite sense.

In a crystal which is only OPTICALLY TWINNED, one portion of the crystal is of OPPOSITE HANDEDNESS, and electrical sense, to another portion. This twinning (but not electrical) is detectable by optical means (polarized light) and is named optical twinning for this reason.

The extent of twinning that may be present in commercial crystals is seen in Fig. 5.1, which shows both electrical and optical twinning boundaries at the top surface of some Z-cut (basal) sections of quartz (which were cut up for the manufacture of quartz oscillators). Though the crystals are seldom entirely free of twinning, they do not on the average run as badly twinned as here shown. These views, taken by means to be described, correspond to what one sees when examining an etched quartz surface by reflection from a strong light.

Since untwinned finished plates must be cut entirely from one twin or another (not across a boundary), and since the proper sense of angular orientation of the plate is opposite for two adjacent electrical twins, the economic utilization of twinned quartz is a difficult problem.[4] It involves cutting the

[3] Etching is also used on finished plates for removing grinding debris, and for frequency adjustment.

[4] As herein used, a *twin* is one of the homogeneous, untwinned portions of a twinned crystal.

stone into separate parts when the twins are large enough to be utilized separately. Further, at some stage before reaching the finished plate all twin portions but one must be cut away.[5]

In this connection it is important to note a size and form difference between electrical and optical twins. Fig. 5.2 shows the appearance of twinning boundaries when only ELECTRICAL TWINNING is present. Note that electrical twins are commonly large, hence may often be separated ap-

Fig. 5.1—Examples of ELECTRICAL and OPTICAL twinning, as exhibited at the etched surface of Z-cut sections. These examples are typical of an appreciable portion of the quartz that is cut up for quartz plates.

proximately along a boundary and both portions utilized. Fig. 5.3 shows the appearance of twinning boundaries when only OPTICAL TWINNING is present. Since optical twins are commonly small and in the form of thin laminations, it is seldom possible to cut optical twins apart and use both parts separately.

The conventions here used, regarding handedness and axial sense, are

[5] See Section 5.7 for the possibility of utilizing partially twinned finished plates.

according to those of the proposed "I. R. E. Standard."[6] Figure 5.4 shows the relation of these conventions to the natural faces of right and left quartz, to the electric charges developed on compression and tension, and to the more common cuts of oscillator plates. Also given are the relations of handedness to the conoscope and the polariscope means of detecting handedness (Section 2.7, Chap. II describes these instruments). It is important to

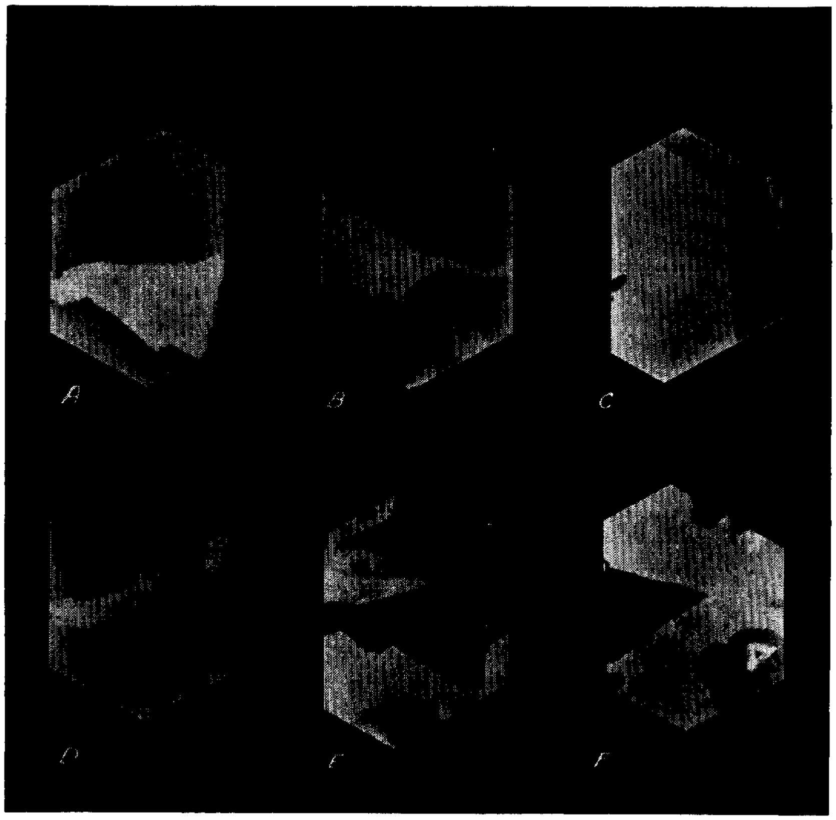

Fig. 5.2—Examples of ELECTRICAL twinning alone. Electrical twins are commonly large, and hence may be cut apart and used individually.

note that AT and CT plates are always cut at such an angular sense, relative to the Z and X axes, as to be roughly parallel to a *minor pyramidal face*, whereas the BT and DT plates are roughly parallel to a *major pyramidal face*. Thus a stone exhibiting these faces may be cut into any of these plates

[6] "Proposed Standard Conventions for Expressing the Elastic and Piezoelectric Properties of Right and Left Quartz", *Proc. I. R. E.*, Nov. 1942, p. 495. Also, I. R. E. "Standards on Piezoelectric Crystals, Recommended Terminology," 1945.

without determining the handedness and electrical sense of the stone (if twinning is negligible). As will be seen later, a similar situation prevails when analyzing etched X-cut sections for cutting into plates.

5.3 Nature of Etch-Pits

When crystal quartz is etched by contact with hydrofluoric acid (or other etching agents) the surface of the quartz is eaten away in such a manner as

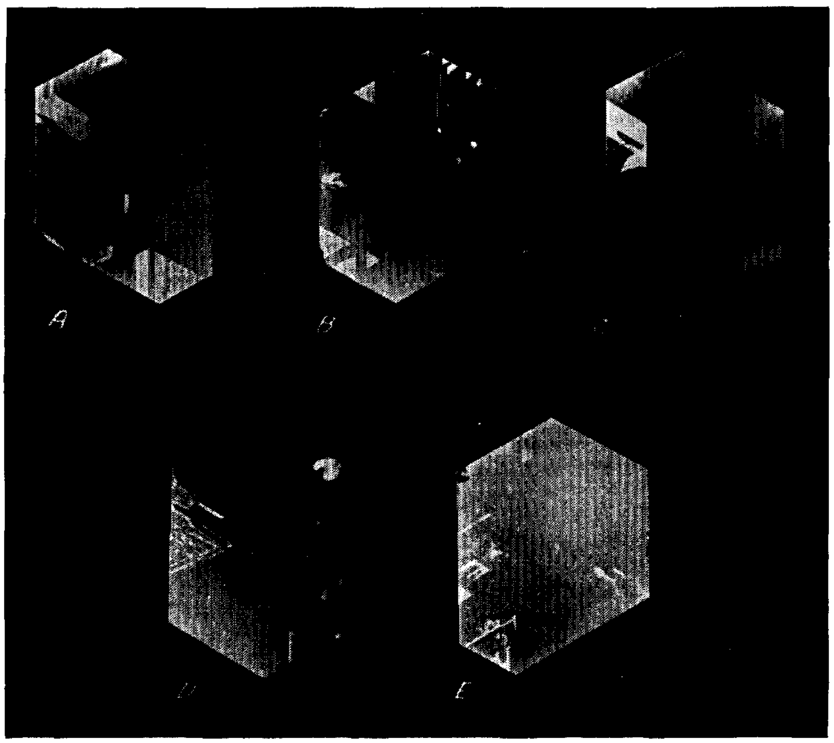

Fig. 5.3—Examples of OPTICAL twinning alone. Optical twins are commonly small and interlayered, and hence may not be separated and used individually.

to leave microscopic *etch-pits* (or hills). These etch-pits are formed of minute facets which are definitely related to the crystal structure. The form of these pits and the orientation of the facets may be used to determine the orientation of the crystal structure at the etched surface being examined.

The general appearance of four types of etch-pits is shown in the photomicrographs of Fig. 5.5. These are the pits that are developed on ground surfaces which are approximately parallel to the well known X-, Y-, and Z-cut surfaces of right hand quartz, by the action of hydrofluoric acid. It is seen that the positive and negative X-surfaces produce different etch-pits, and are thus usable in determining electrical sense. Further, the pits on all surfaces have directional properties which allow them to be used for determining the approximate directions of the axis which lie in the etched surface. However, to be able to determine orientations from etched surfaces of other

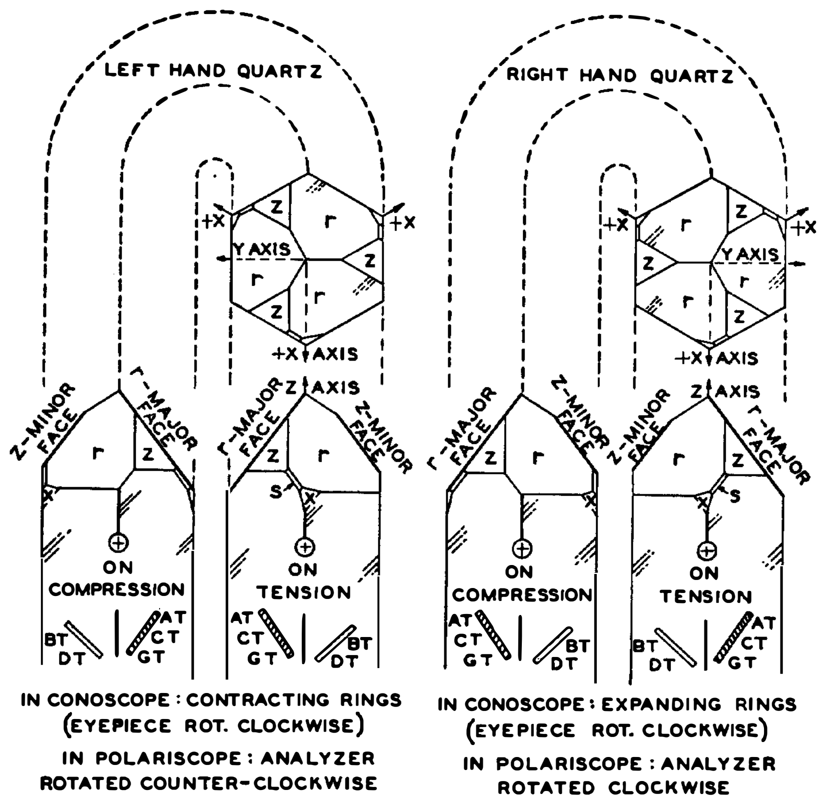

Fig. 5.4--The conventions of handedness, axes, natural faces, and angular sense-of-cut of common oscillator plates, together with the electrical and optical rules for determining these characteristics in unfaced stones.

orientations than those shown above, requires a knowledge of the appearance of the etch-pits developed on such surfaces.

A rather complete catalog of etch-pits on all possible surfaces of quartz was prepared by W. L. Bond,[7] using an etched sphere of quartz (Figs. 5.5, 5.6 are from Bond). Thirty-six different types of etch-pits were obtained and their angular range of coverage was found (the X-, Y-, and Z- surface

[7] "Etch Figures of Quartz," *Z. Kristallogr.* (a) 99, 1938, pp. 488–498.

pits are obtained only on surfaces within 6° to 8°, from the X-, Y-, and Z-surfaces, respectively). Since the development of good etch pits and their exact appearance is considerably affected by the preparation of the surface for etching (fineness of grind), and by the strength of the acid and the length of etching time, and by the manner of illumination when viewing, the

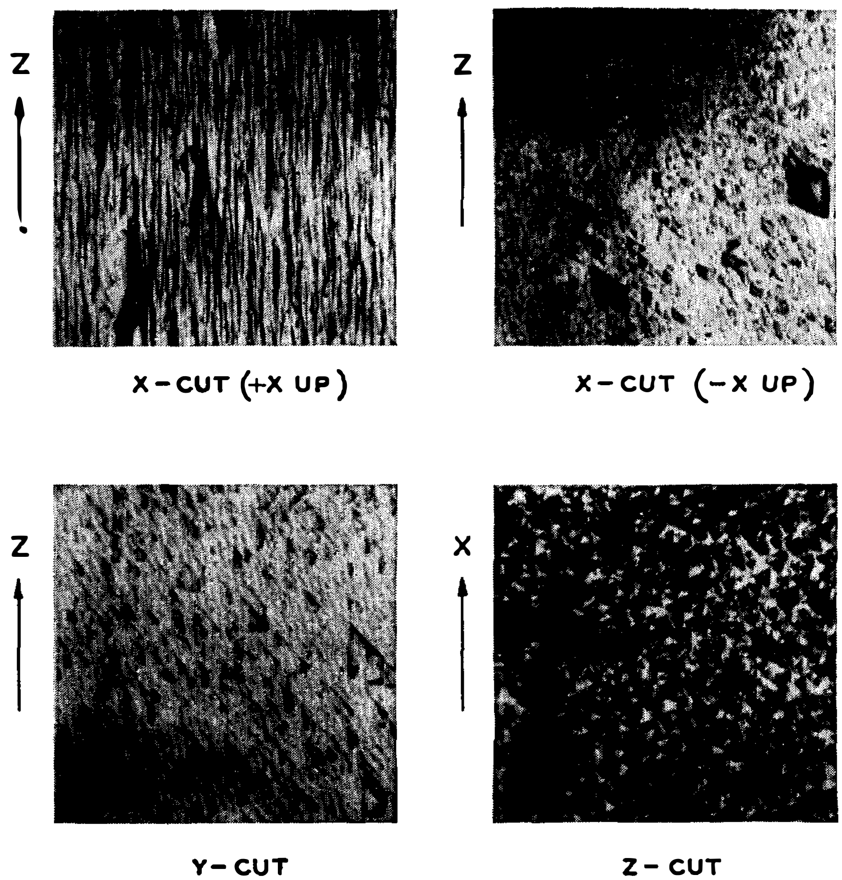

Fig. 5.5—Photomicrographs of etch-pits on the etched surfaces of common orientations. As seen the etch-pits are definitely related to the structure axes of the quartz.

figures shown here do not represent the exact appearance of pits obtained by other manners of development. However, such figures are reproducible.

The use of etch-pits to determine the orientation of a perfectly general surface is complicated by the fact that some different surface orientations give pits not readily distinguished from each other. However, for the surfaces most commonly encountered in quartz plate manufacture the etch-pits are quite distinctive, when well developed. Use may be made of a microscope or a high powered projector to view the figures. The pit outlines may be aligned with lines ruled on the eye-piece or on the screen, and a fixed marking device may be used to mark the quartz surface with orientation lines. Twinning may be detected by the appearance of different etch-pits as the specimen is moved about. For example, on an electrically twinned X-cut surface both X-cut views of Fig. 5.5 could be found. However, the location and marking of twinning boundaries involves a tedious exploration of the surface, since only a minute portion is viewed at any one time. This exploration may be eliminated if the surface is first viewed by reflection methods where the whole surface and extent of twinning is at once seen (as in Fig. 5.1) and marked.

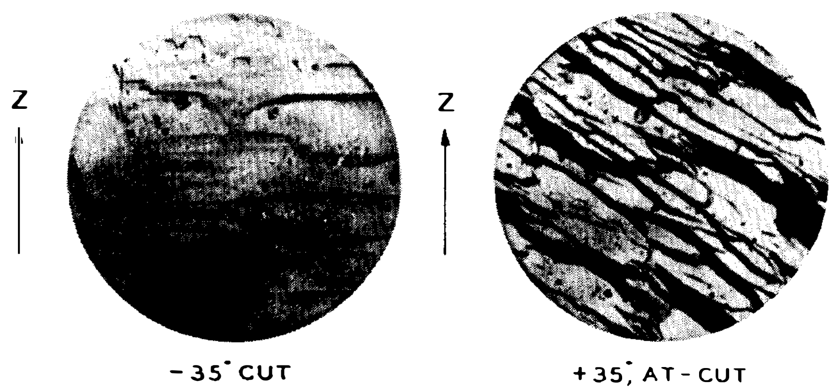

Fig. 5.6—Etch-pits on the etched surface of a +35° AT plate, and on an analogous but wrong sensed −35° plate. This difference in etch-pits may be used in the manufacturing process to determine the right and wrong sensed regions of twinned AT slabs.

A special case where the microscope or projector method might be employed is in the examination of thin AT, BT, CT or DT slabs for twinning and sense of cut. Here the slabs are known to be cut with a reference edge parallel to an electric axis, and with the major faces inclined at 35° to 55° (depending upon the variety of slab) from the optic axis, the sense of the inclination being positive for the AT and CT slabs, and negative for the BT and DT. The effect of electrical twinning on such etched surfaces is shown in Fig. 5.6. The etch-pits of the good +35° AT-portion of the slab are easily distinguished from the analogous −35° (bad) portions. This difference is similarly distinguishable in the other cuts.

Actually, orientation and twinning are seldom analyzed by the method described above, i.e. by examining their appearance in the microscope, or by projection on the screen. The method appears to be far less practical than other methods which depend upon the gross effect, of hundreds of similar etch-pits, in bending a light beam. By the latter methods the individual etch-pits are never seen, nor does their nature need to be known. Nevertheless, the resultant optical effect of hundreds of similar etch pits is as characteristic of structure orientation as the individual pits themselves.

5.4 Optical Effect of Etch-Pits

The gross optical effect of hundreds of similar etch-pits results from the fact that each of the pits has minute facets which are similarly inclined to those of all the other pits. Though the pits of Figs. 5.5 and 5.6 may not appear to be formed from groups of flat facets they are generally so regarded. "Curved-facets" are theoretically considered to be made up of individual flat-facets which are parallel to possible atomic planes (and hence may be given index numbers as in Chap. III). This view is the same as that taken

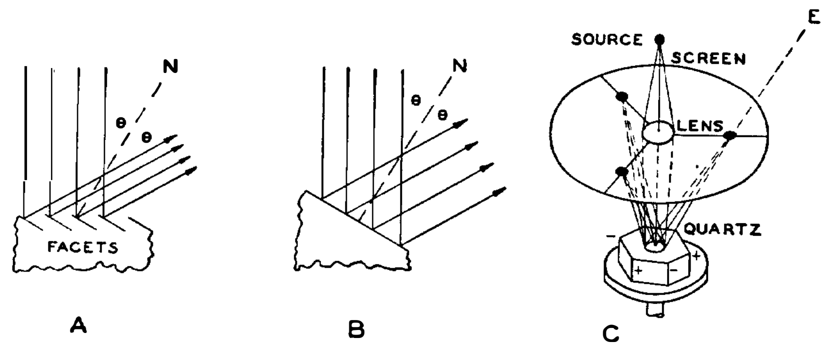

Fig. 5.7—Reflection of light from a single set of similarly oriented etch-pit facets, A, is like that from a single mirror, B. Reflection from all three sets of facets of a Z-cut section will give a three-fold *etch-figure* on a screen, as in C.

with regard to natural faces, which are of course produced by essentially opposite effects, i.e., acid corrosion in the case of etch-pits, and growth from solution in the case of natural faces. Actually, many "curved-facets" give optical effects showing no discernible evidence of individual flat facets. However, the question is academic, so far as use of the pits for orientation purposes is concerned, for such facets are still definitely related to the crystal structure.

Etch-pit facets may be used to *reflect* a light beam into specific patterns or to *refract* the beam on transmission through the material into similar (but not identical) patterns. The different basic optical means of using etch-pit facets are shown in Figs. 5.7, 5.8, 5.9. Included in each figure is a diagram of the effects obtained by illuminating an idealized Z-cut section. This idealized section is assumed to have only simple, equilateral, three-sided pyramidal etch-pits, oriented relative to the X axes as shown in Fig. 5.5. The actual results obtained with Z sections are more complicated than this and thus indicate that the etch-pits are not exactly as idealized here.

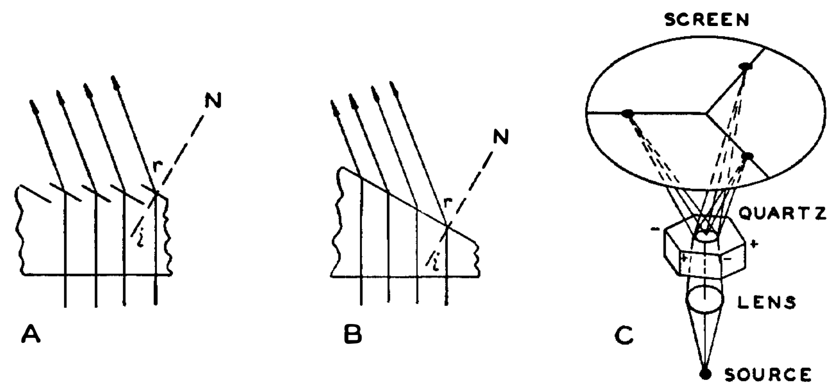

Fig. 5.8—Light transmitted thru a single set of etch-pit facets, A, is refracted as by a prism, B. The three sets of facets of a Z-cut section give a three-fold etch-figure, as in C.

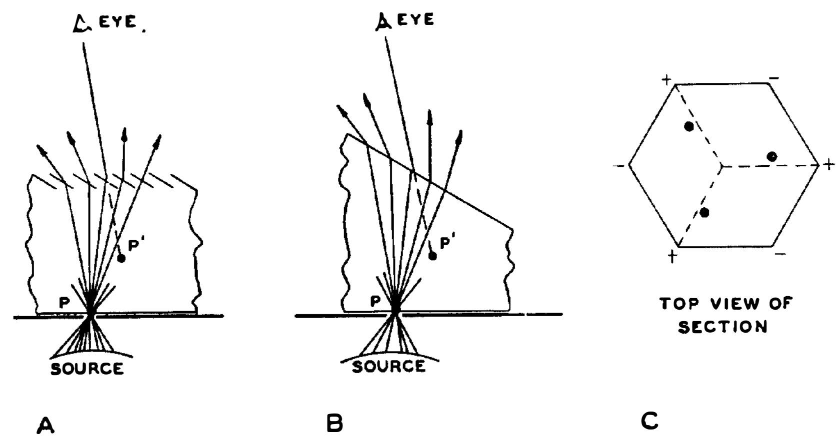

Fig. 5.9—Light transmitted thru a pin-hole is refracted by a single set of facets, A, as it would be by a prism, B. A virtual image of the pin-hole P will be observed at P′. The etch-figure seen down in a Z-cut section is three-fold, as in C.

5.41 The Reflection Method

Figure 5.7 shows the reflection method, where a parallel beam of light striking the etched surface of a Z-section is reflected from one of the three sets of facets as shown in A. Each single facet reflects part of the beam by ordinary reflection laws, and the whole groups of facets act similarly to a single mirror surface at the same angle, as in B.[8] The individual facets being very minute and of irregular size and spacing, however, cause appreciable diffusion of the beam. The resultant effect of all *three* sets of facets is shown in C, where light passing down through a lens and a hole in the screen is reflected back to three spots on the screen. These three spots are located at equal distances from the incident beam and at 120° intervals around the incident beam. If the quartz section be rotated on its table the spots rotate around the screen correspondingly. However, lateral motion of the section across the table (without rotation) does not change the position of the spots, if the section be untwinned. If the section is twinned (or more exactly, if the etched surface is twinned) the three-fold figure will shift to a different position (angularly) on crossing a twinning boundary, for the etch pits are oriented differently in the two twins. If the twinning boundary divides the illuminating beam, then both figures appear at once, giving six spots instead of three. It is clear then that twinning, as well as orientation of the section, may be determined from the figure on the screen. The angular relation between the spots and the X-axes of the section will be considered later, where figures of actual sections are shown.

The long used method of examining etched quartz surfaces by simple reflection from a bright light, may also be explained from Fig. 5.7C. If a spot of light on the screen is viewed along the line E, and the screen then removed, the light from the associted etch-pits will fall on to the eye. The illuminated portion of the section will appear bright. If a twinning boundary crosses the illuminating beam and one of the six reflected beams falls on the eye, one of the two illuminated twins will appear bright and the other dark. As the section is rotated, first one twin and then the other will appear bright, and in each case the twinning boundary is sharply defined over the whole region covered by the illuminating beam (the appearance of twinned Z-cut surfaces examined by this means is shown in Figs. 5.1, 5.2, 5.3). Due to the greater complexity of etch-pits than here idealized, the reflected beams are not so sharply defined as to require exact location of the eye relative to the incident beam and the section. Further, when a broad unfocused light source is used, it is possible and convenient to detect twinning boundaries merely by holding the section in the hand and rocking it about in various directions until a brightness contrast is observed. Though the brightness contrast is usually not marked by this simple examination it suffices for many purposes.

[8] That the effect of a group of facets is not identically the same as that of a single mirror, is of more concern where lenses are used for focusing. In this case the displacement of the mirror facets causes a displacement of the focus of the beam from each facet. For beams of small angular range this is of little importance.

5.42 The Transmission Method

Figure 5.8 shows one form of the transmission method of examining Z-cut etched surfaces. A parallel beam of light passing normally up through the bottom polished surface and the top etched surface of a section will be bent by refraction only at the etched surface, as in A. Each facet refracts the light by ordinary laws of refraction, and the whole group acts similarly to a single refracting surface at this angle, as in B.[9] The resultant effect of all three sets of facets is shown in C (where a lens is added for focusing the light beam). If the incident beam is not normal to the bottom surface there is an additional bending of the beam at this surface. If the incident surface is not polished (or rendered optically flat, with a cover glass and immersion fluid, for example) the diffusion at this surface will mask or completely destroy the desired effect.[10]

5.43 The Pinhole Transmission Method

Figure 5.9 shows the pinhole form of the transmission method, as applied to the examination of Z-cut etched surfaces. Here a section with a top, etched surface is illuminated from below through a small hole with a wide angle of illumination. The light radiates upward in all directions from the pinhole, and in passing through the upper etched surface is refracted by a single set of etch facets as in A. With the eye placed above the pinhole (and section), certain of these rays will fall on the eye. The eye then sees a virtual image of the pinhole P displaced to P′, elevated from the level of P, and along the line of the ray which enters the eye. The effect of a group of facets is similar to that of a single prism, as in B.[11] The resultant effect of all three sets of facets of a Z-cut section is shown in C, where the section is viewed from directly above and no optical system is shown. Only the three virtual images of the pinhole are seen and they are located down in the quartz (roughly two-thirds of the way down).

Though the desired effect is due entirely to the top, etched surface, the nature of the bottom surface may cause a deleterious masking effect, which must be considered in the design of an instrument. Due to the diffusing effect of irregularities in the top surface it may act somewhat as a screen upon which the extended light source shown in Fig. 5.9A, B may be imaged by the pinhole. This extraneous image occurs if the bottom surface is polished, and to some extent if the surface is semi-polished, strongly etched, or oily.

[9] See footnote 8.

[10] Similar optics hold if the section is illuminated from the etched side instead of the polished side.

[11] See footnote 8.

This difficulty may be entirely obviated by the introduction of a diffusion screen directly adjacent to the pinhole.[12]

It might be noted that if it be desired to project or photograph the pinhole figure, one must focus on the virtual image which lies between the top and bottom surfaces of the etched specimen. In the simple case diagrammed in Fig. 5.10, it is assumed that the camera lens is at a distance from the section and directly over the section, so that the rays to the lens are essentially normal to the section. For a section of thickness T, and index of refraction n, the elevation E of the virtual image from the bottom surface of the section is given by: $E/T = 1 - \sqrt{1 + R^2/T^2}/n$. Here R is the radial displacement of the virtual image from the axis of the pinhole and is readily observed and measured. Also, R may be calculated from the thickness of the quartz T, the angle θ between the facets and the gross surface, and the in-

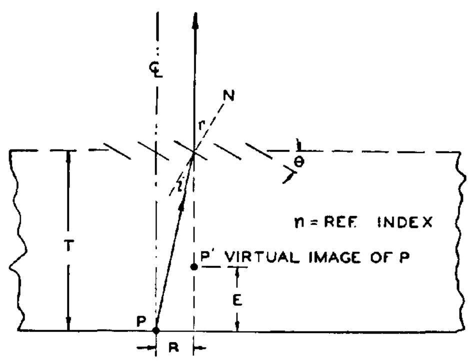

Fig. 5.10—The elevation E of the virtual image may be calculated from the thickness of the etched section T, the radial displacement of the image R, and the index of refraction n; or from T, n, and θ, the angle between the facets and the gross surface.

dex n, (or θ may be calculated from T, R, n) by: $R/T = \tan(\theta - \sin^{-1}[(\sin \theta)/n])$. Commonly, pinhole figures from quartz which is weakly to moderately etched (up to one hour in concentrated HF) have a maximum diameter (or double radial displacement) $2R$, nearly equal to the thickness of the section. Since the elevation of the image, E, depends upon its displacement R, an extended virtual image is not in a single plane and cannot be exactly focused (the elevation is commonly about one-fourth to one-third of the thickness of the section). The diameter of the pin-hole must always be kept small compared to the thickness of the section to give sharp figures (and the length of the pinhole must be small compared to its diameter).

[12] The diffusion screen may be a sheet of white paper placed over the pinhole, or a piece of flashed glass placed under the pinhole, with the flashed side against the pinhole. In either case it is usually necessary to increase the light intensity by focusing a concentrated light source onto the pinhole with a lens.

Choice of one of the four above methods of examining etched surfaces for twinning and orientation, depends upon many factors, as will be noted in the following section. The pinhole method is used wherever possible because of the simplicity of the optical system and the brilliance of the figures obtained.

5.5 Etch-Figure Instruments

Herein are described several instruments which have been designed for shop use in determining orientation and twinning of etched quartz sections and slabs. Their basic principles of operation are as described above. The nomenclature of handedness, sense of axes, sense of cuts, natural faces, etc. is according to Fig. 5.4, as explained at the end of Section 5.2.

The etch-figures and reflection patterns obtained on these instruments vary with the preparation of the specimen (i.e. the type of grind and the type of etch). A complete study of these factors would include a variation of the grind from a very coarse grind to polishing (and include saw-cut surface), and a variation of the etching time from short to very long, and the strength and kind of etching agent. Here chosen for illustration are the simplest practical preparations, namely, the coarsest grind usable, and the shortest etching time (in hydrofluoric acid). The etch-figures are thus markedly different than some which have appeared in the literature. Further, the photographic reproduction of etch-figures on paper, is not exact due to the limited contrast range of the paper. Thus in the accompanying illustrations detail is lost in the brilliant portions of the etch-figures in order to show details in the weaker portions, and vice-versa.[13]

5.51 The Reflection Oriascope

Fig. 5.11 shows diagrammatically a reflection "Oriascope", which may be used on specimens with a single flat etched surface. By the reflection principle of Section 5.41 figures are obtained on a viewing screen. Due to the relatively weak figures obtained by reflection from weakly etched surfaces, the viewing screen must be enclosed in a well blackened enclosure, and viewed through an eye chute. The screen is ruled with appropriate lines, relative to which the figure is aligned by turning the specimen on the table. The table is mounted so that when the specimen is properly oriented, the table may be slid to the right or left over a marking template, and marked through the template with appropriate lines to indicate the desired axial orientations of the specimen.

When used with Z-cut sections it is necessary to have two marking templates, one for each handedness of the quartz, since the three-fold figures obtained are not aligned with the electric axes of the specimen. They are

[13] Apparent shifts in etch-figure orientation, with etching time for example, are not to be considered as resulting from an orientation shift of the individual etch-pit-*facets*, but as a shift in the relative areas of differently oriented facets. See Figs. 5.12 and 5.17.

shifted approximately 12° therefrom, and in opposite directions for the right and left varieties. Figure 5.11 shows a section of right quartz so positioned on the sliding table that the etch-figure therefrom will be properly aligned with three radial lines of the viewing screen. The section need not have natural faces as here shown. With the section so positioned the sliding table is moved over the right-hand marking template, and the section is marked with three radial lines. These lines on the section then give the approximate direction (within 5°) and the sense of the three electric axes of the quartz, positive X-outward. With left quartz the etch-figure is still aligned with the same lines on the viewing screen, but the section is marked through the left-hand marking template (the marking having the same meaning as be-

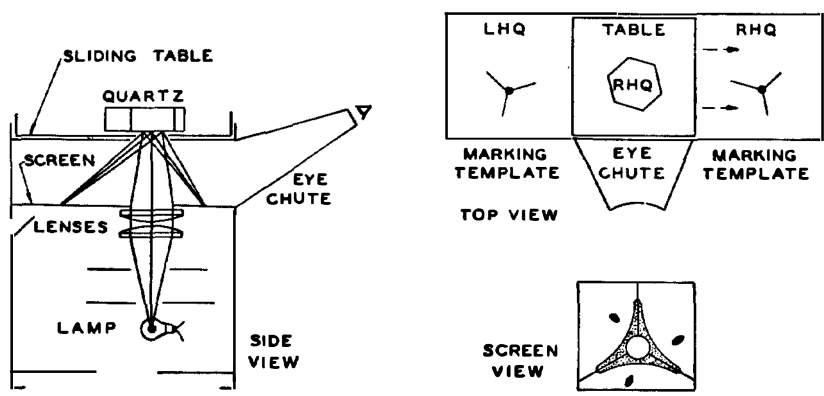

Fig. 5.11—The reflection ORIASCOPE as applied to determining the direction and sense of the X (electric) axes in Z-cut sections. After the etch-figure is aligned on the screen the table and sections are moved over a marking template and the section marked from below with axes.

fore). The section so marked is ready for laying out the approximate cutting directions, the sense of which may be found from Fig. 5.4. The exact cutting directions are obtained by X-rays. It might be noted that ordinarily the handedness of the section is determined in the conoscope (see Section 2.7, Chap. II) before examination on the oriascope. Also the twinning boundaries are previously determined by examination of the etched surface in a spot-light beam.

Figure 5.12A, B show the type of etch-figures obtained on Z-cut sections (in each case the figure is properly aligned with the rulings on the viewing screen). The simpler etch-figure A is obtained on a fine ground (400 carborundum) surface by a weak etch (about 10 minutes in 50% HF). Though the three faint spots, about 40° clockwise from the rulings (for the left-hand quartz of A) may be used for determining the handedness of the section, it is usually considered more reliable to use the conoscope for handedness determination. The counter-clockwise rotation of these spots in B indicates right-hand quartz. The more complicated etch-figure B, results from etching a fine ground surface too long,[14] or from using a coarse instead of a fine grind. With such figures it is difficult to know which portion of the figure is to be aligned with the screen rulings. Hence the sections must be fine ground and the etching time closely controlled.

The obvious disadvantages of the reflection oriascope (the necessity of predetermining handedness and twinning, and the requirements of fine ground surfaces and closely controlled etching time) are largely overcome by the pin-hole oriascope, later described. However, the reflection oriascope is an

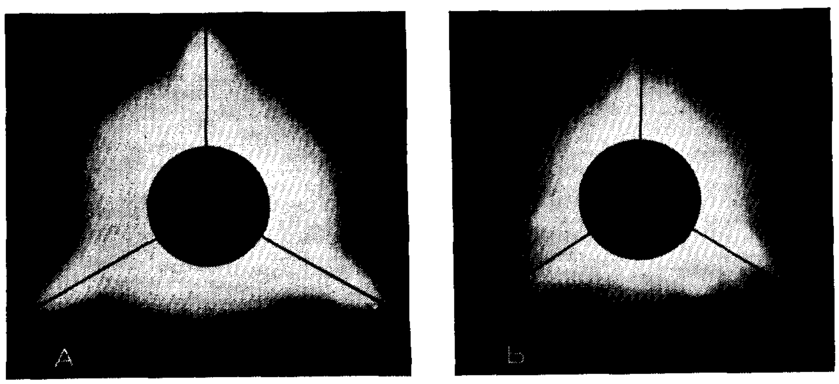

Fig. 5.12—Etch-figures obtained on the reflection oriascope with Z-cut sections (reduced from 11 inches square). A is a good usable figure while B is difficult to use due to its complexity.

excellent explanatory instrument for obtaining experimental etch-figures from surfaces of any orientation, preliminary to devising a special instrument to most advantageously utilize the reflection characteristics found. This fact results from the large and symmetrical screen coverage, and from the fact that only one etch surface is encountered by the light beam (thickness and back surface shape is of no concern).

5.52 The Reflection Twinoriascope

Figure 5.13 shows diagrammatically a reflection "Twinoriascope" designed especially for shop use in detecting and marking twinning boundaries and the sense of orientation in etched AT, BT, CT and DT slabs. When, for example, CT slabs are to be examined the tiltable mounting-table is clamped in the 38° position, and the slab placed crosswise on the table (X-axis normal

[14] It appears that excessively strong etches (hours long) again give a simple, strong, and reliable figure.

to line of sight, and beveled edge as shown). Upon moving the viewing screen to position *1*, only lamp *1* is lighted, and the slab is viewed by reflected light at a preferred angle. If the slab be twinned, one portion of the slab will exhibit a bright sheen while the other portion is dull by contrast, see two examples in Fig. 5.14, Test *1*. The twinning boundary is now penciled in. The viewing screen is then shifted to position *2* which lights only lamp *2*, and the crystal moved to right or left so that only one twin is illuminated. On the screen[15] will be seen an etch-figure similar to one of the four shown in Fig. 5.14, Test *2*. If either of the two positive-cut figures are observed the illuminated portion of the slab is usable, since the CT plate must have a positive 38° orientation. The negative-cut, "golf-club", figures are produced by the unusable portion of the plate.

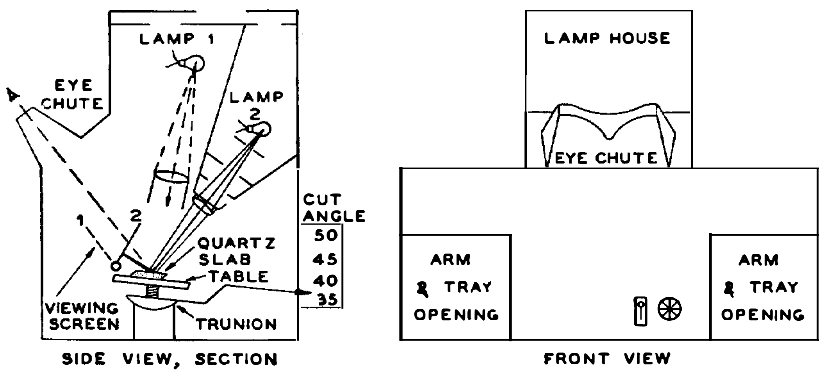

Fig. 5.13—The reflection TWINORIASCOPE for detecting *twinning* (using lamp *1* and no viewing screen, position *1*) and for determining the orientation or *sense-of-cut* (using lamp *2* and the viewing screen in position *2*), of AT- BT-, CT-, or DT-cut slabs. The "cut angle" is set for a CT slab.

The same procedures are followed with the AT, BT and DT plates, in each case resetting the table to the proper tilt, 35°, 49° and 52°, respectively. The reflection view of Test *1* is the same for all cuts, and the etch-figures of Test *2* are nearly the same (being almost identical for the negative-cut portions of the slabs). However, in the case of AT and CT slabs the *positive-figures* represent *good* portions (since these are positive cuts), and in the case of BT and DT slabs, the *negative-figures* represent *good* portions.

The basic principle of this instrument is as described in section 5.41. As here used, the two optical systems (including the eye and the slab) are so disposed as to obtain the best reflection-contrast in Test *1*, and the most distinct portion of the etch-figures in Test *2*. That the observations are so similar for this 20° range of cuts indicates that the nature of the etch-pits

[15] An excellent screen consists of two sheets of thin sandblasted cellulose acetate.

on these cuts is very similar, (see Fig. 5.6 for the nature of the etch-pits on AT slabs). The angular arrangement of the Test *1* optical system makes use of strongly developed facets which are approximately parallel to the X-axis and inclined at an angle of $-57.6°$ to the Z-axis of the quartz. Within experimental error these facets are parallel to the 01.2 atomic planes and hence are called the 01.2 facets. It is also these facets that give the enlarged

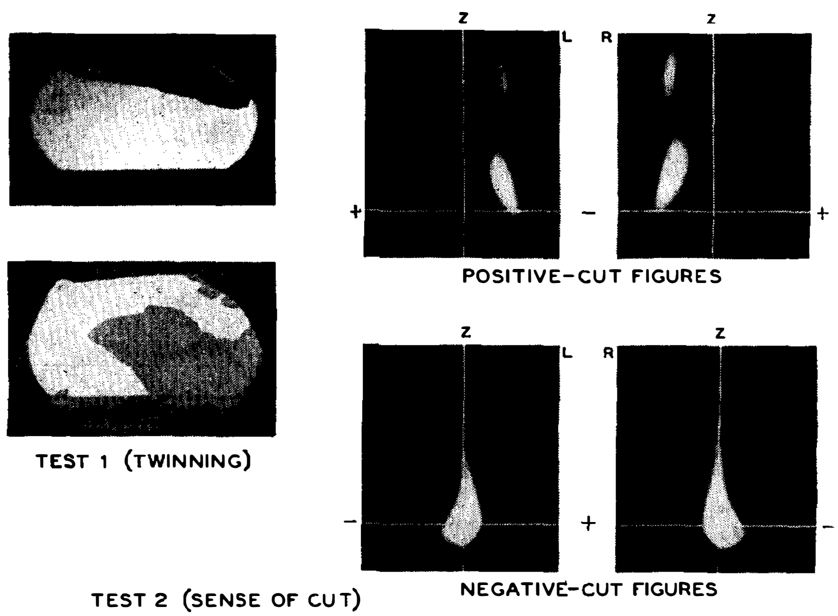

Fig. 5.14—The appearance in the twinoriascope of twinning in Test *1* (two examples) and of the four possible etch-figures in Test *2*. The observance (in Test *2*) of either of the positive-cut figures indicates that the illuminated portion of the slab is a positive cut, while either negative-cut figure indicates a negative cut. These etch-figures for a CT slab, are not markedly different than those for AT, BT, and DT slabs.

head of the golf-club, negative-cut figures. The right and left handedness of quartz results in two figures each for the positive and the negative orientation. Though it is commonly of no interest, it is possible to determine from the etch-figure observed, both the handedness and the electrical sense of the illuminated portion of the slab. The handedness is as indicated by L and R in each etch-figure of Fig. 5.14, and the electric axis is $\pm$ to the right or left as indicated by the $+$ and $-$ signs.

Best etch-figures are obtained in the twinoriascope with fine ground (400 carborundun) slabs which have been given a strong etch (40 minutes in 50% HF). Stronger etching is not deleterious. Very strong etching gives moderately good figures with sawn or coarse ground slabs. For Test *1*, alone, weaker etches would suffice. Under properly controlled conditions of slab preparation and instrument operation Test *2* might be eliminated, for under such conditions the negative-cut portion of the slab is bright, the positive-cut portion is dark. Under shop conditions this means of detecting *sense* of cut appears to be not reliable, especially with *untwinned slabs* (which are either all bright or all dark). The addition of Test *2*, however, gives

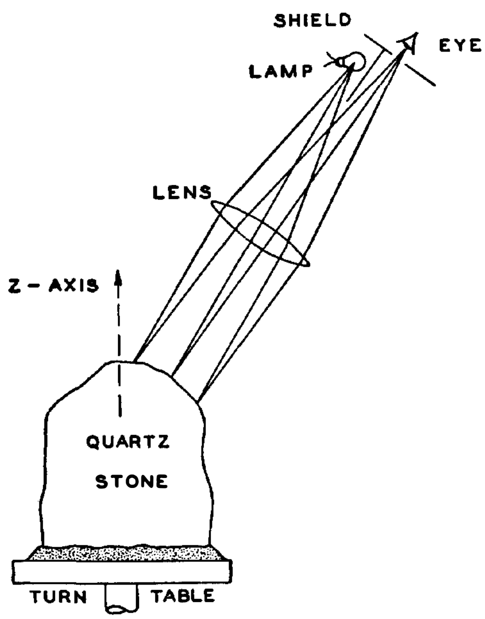

Fig. 5.15—The direction and sense of the electric axes of a sand-blasted and etched raw quartz stone may be determined by reflection of light from the 0.21 facets. These same facets are utilized in Test *1* of the twinoriascope, Figs. 5.13, 5.14.

complete reliability, for if etch-figures are obtained the sense of cut is obvious, if no figures are obtained the slab can be returned for further etching.

The principle of Test *1*, above, has been applied by W. L. Bond to a laboratory instrument for determining the direction and sense of the X-axes in raw quartz stones prepared with a sand-blasted and etched surface. With the stone mounted rotateably about its Z-axis (previously determined by conoscope or inspectoscope), and a light beam properly projected onto the stone, reflection of the light beam to an eye piece or viewing screen will occur whenever the 01.2 facets come into proper angular position, see Fig. 5.15. The approximate direction and sense of the electric axis, or the sense of cuts to be made from the stone, may be determined from these reflecting positions of the stone, and twinning may be partially explored. Thus if the stone appears to be not badly twinned, it may be cut up at once into slabs of proper sense of cut, without previously sectioning for further examination.

5.53 The Pin-Hole Oriascope

Figure 5.16 shows a "Basic Pin-Hole Oriascope" with auxillary attachments for shop examination of etched Z-cut sections, and Fig. 5.18 the same

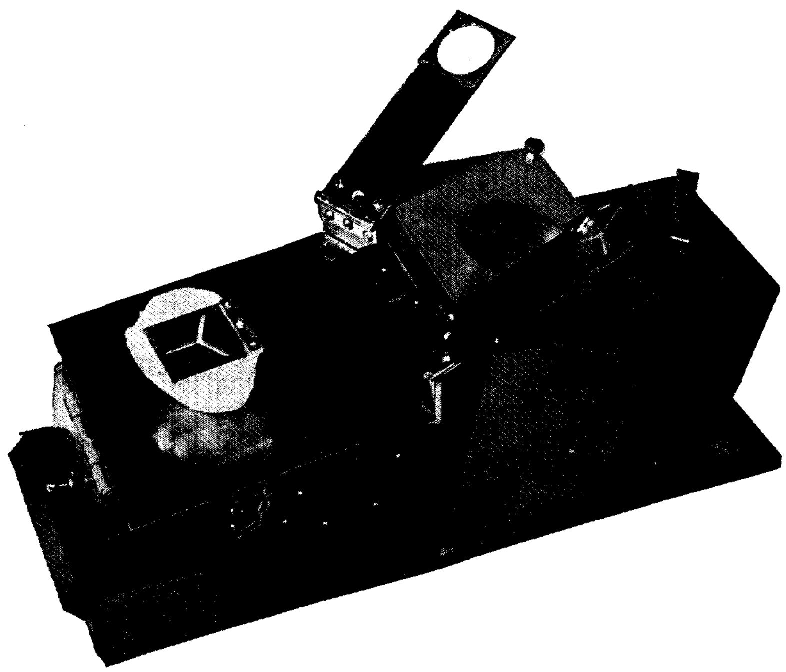

Fig. 5.16—The BASIC PIN-HOLE ORIASCOPE with matching and marking arms for use on Z-cut sections. Twinning, and the direction and sense of the X (electric) axes may be determined and marked on the section.

for X-cut sections. The optical principle of this instrument is according to Section 5.43. Light from a concentrated-filament lamp within the central ventilated housing, is projected horizontally forward by a pair of condenser lenses and reflected upward by a mirror in the forward housing, onto a diffusion-disk placed directly against the pin-hole.[16] The latter is centrally located in the inclined mounting table. Etched quartz sections are placed over this pin-hole and viewed from above. The section may be moved about and examined for twinning boundaries, which are then penciled in.

[16] See footnote 12.

The section is then examined through the ruled window of a matching arm, one of which is shown in use in Fig. 5.18. The section is rotated on the table until the etch-figure seen in the quartz is properly aligned with the lines on the window. Without moving the sections, the viewing arm is replaced with a marking arm, one of which is shown in place in Fig. 5.16. The section is

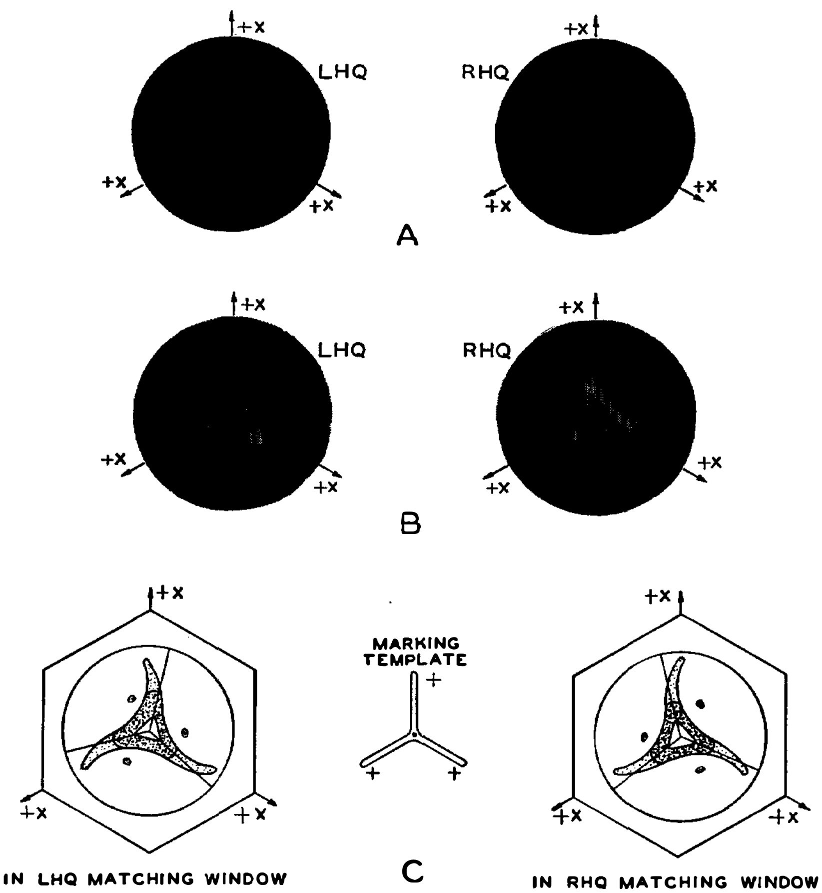

Fig. 5.17—Etch-figures obtained with the pin-hole oriascope in Z-cut sections; A for a fine ground surface and B for a coarse grind. The relation of the etch-figures to the structure orientation of the section is shown in C.

marked through the template of this arm with the desired axes or cutting directions.

Figure 5.17A, B shows the etch-figures obtained with the pin-hole oriascope, on Z-cut sections. Figure 5.17A is for a fine ground surface (600 carborundum) while Fig. 5.17B is for a coarse ground surface (100 carborundum), and in both cases a moderate etch, (20 to 30 minutes in 50% HF). It is noted that the spiralling, outer tails of the etch-figures (as well as other features) denote the handedness of the quartz. Such handedness features are not as marked with fine ground surfaces, nor with weaker etches. The central triangular portion of these figures is used for alignment of the section with the rulings on the marking arm windows. Since this triangular figure is misaligned with the X-axes of the quartz by approximately 12°, and in an opposite sense for the two kinds of handedness, there are provided two matching arms. One is to be used for left quartz and the other for right quartz. The diagram of Fig. 5.17C shows the orientation arrangement of a combination of matching windows and marking template, that results in the section being marked with three radial lines which correspond to the positive X-axes of the quartz. Though this is the most obvious manner of marking Z-cut sections, it is of advantage in practice to obtain a reversed marking on left-hand quartz (by using an oppositely ruled left-hand matching window). By so marking the quartz no further attention need be paid to handedness, see Section 2.4, Chap. II.[17] In either case the relation of the various plate cuts to the axis markings obtained above, may be determined from Fig. 5.4. Since the etch-figures give only approximate orientation X-rays are used for the final determination. That X-rays are not used for the whole determination is as explained in Section 5.1.

With X-cut sections, having a coarse grind (100 carborundum) and a strong etch (30–45 minutes in 50% HF), the etch-figures obtained are like those of Fig. 5.19. Here the positive face of the section gives an entirely different figure than the negative face, as would be expected from the nature of the etch-pits shown in Fig. 5.5. Opposite-handedness gives reversed figures. The four possible figures are oriented with respect to the Z-axis and the major cap face direction of the section "r" as shown in Fig. 5.19A and B. The non-parallelism of the Z-axis and the parallel sides of the etch-figures amounts to three to five degrees. This disposition of figures (relative to quartz axes) is taken into account in the design of the matching and marking arms shown in Fig. 5.18, and diagrammed in Fig. 5.19C. The etched X-cut section is rotated on the mounting table, with the central matching arm in position, until the long straight sides of the "parallelogram" figure, or the long parallel lines of the "H" figure, are parallel to the two parallel-lines ruled on the window of the matching-arm (the parallelogram figure is shown so aligned in C). The figure thus used is compared with the four figures sketched on this matching-arm, to determine which of the two marking arms is to be used for marking (note arrows giving this indication). The proper marking arm is lowered onto the section and used to

[17] The instrument of Fig. 5.16 has a still different arrangement of matching and marking arms.

mark a long line approximately parallel to the optic axis and a short line indicating, in the case shown, the approximate direction and the sense of cut of a BT-plate. It is to be noted, here, that neither handedness nor electrical sense need be individually determined or considered, as such, for the sense of cut is directly obtained.

The size of an etch-figure depends upon the thickness of the section being examined, as explained in Section 5.43. For the etch-figures here presented the size of the figure relative to the thickness of the section, may be estimated

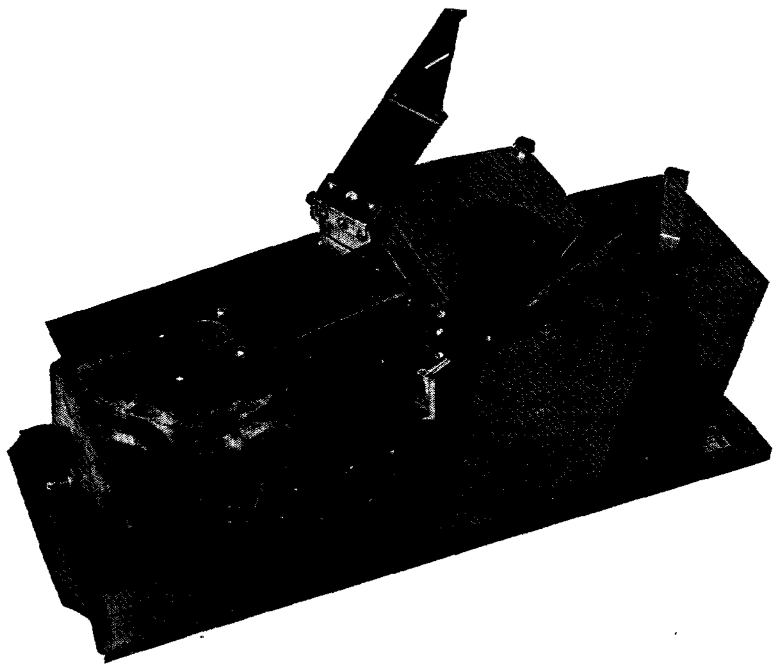

Fig. 5.18—The BASIC PIN-HOLE ORIASCOPE with matching and marking arms for use on X-cut sections. Twinning, and the direction of the Z axis, and the direction and sense of cut may be determined and marked on the section.

from a knowledge of the ratio, N, of the total diameter of the view to the thickness of the section giving that view. For Fig. 5.17A and B, $N = 1.3$; for Fig. 5.19A and B, $N = 2.7$; for Fig. 5.20, $N = 1.7$; for Fig. 5.21, $N = 2.5$.

The pin-hole oriascope may be used in a variety of other ways for examining any crystal cut with at least one etched surface. When used with sections as described above the bottom flat surface may be very small, just large enough to cover the pin-hole. However, this restricts the inspection to an area directly over the bottom surface. This restriction may be eliminated, and no flat bottom surface need be used at all, if the bottom surface

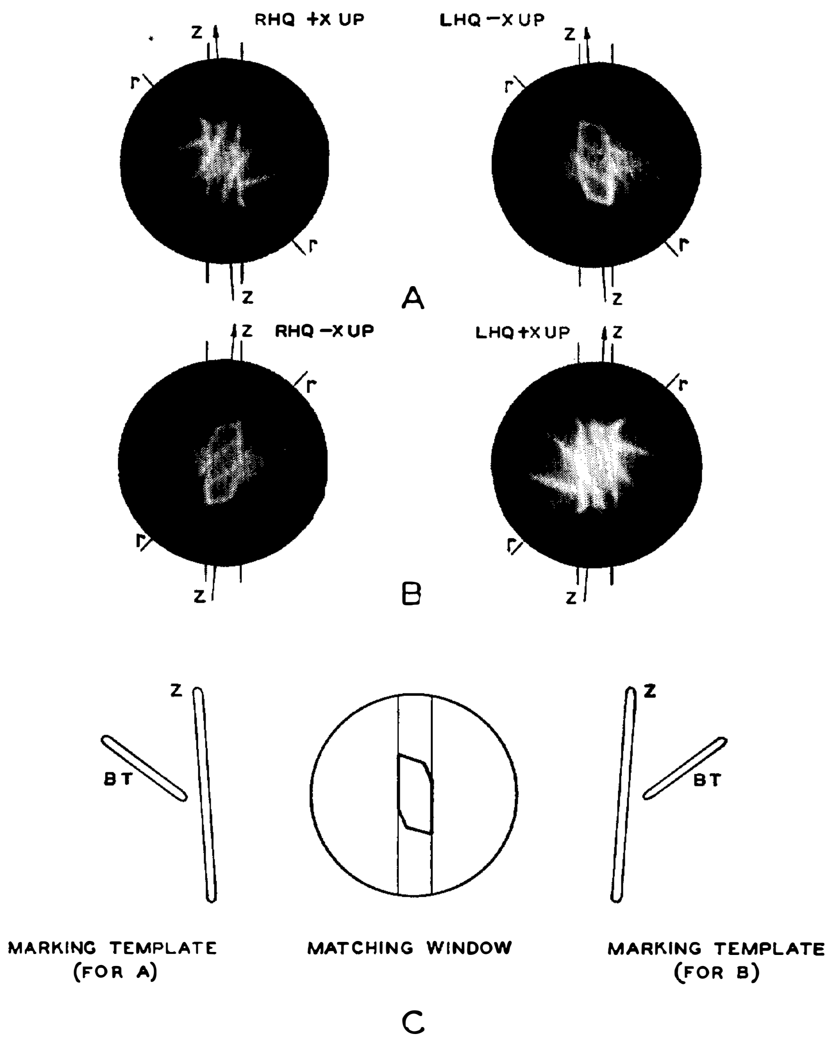

Fig. 5.19—Etch-figures obtained with the pin-hole oriascope in X-cut sections. After an etch-figure is aligned with the rulings on the matching window, as in C, the section is marked thru a marking template (in this case the one on the left) with the direction of the Z axis and the direction of cut of the desired plate (in this case the BT).

of the section be immersed in a transparent dish of immersion fluid (whose refractive index matches that of quartz) placed over the pin-hole. Here the size of the etch-figure depends on the whole distance from the pin-hole to the etched top-surface, and hence, may be made as large as desired, by raising the section and fluid level. Very thin sections, slabs or plates may be examined similarly, with the bottom surface contacting the immersion fluid, or the plates may be wet with immersion fluid and placed on thick glass plates and placed over the pin-hole. In either case the top etched-surface must be kept dry. By this means the twinoriascope examinations described in Section 5.52 might be performed on the pin-hole oriascope, (a disadvantage being the necessity of using an immersion fluid).

Usually etch-figures are obtained from flat etched surfaces whose orientation is known within 5°. However, if the surface be 10° to 20° off-orientation the etch-figure will be plainly distorted. If now the section be viewed at an angle to the normal position, or if the section be tilted in the fluid-

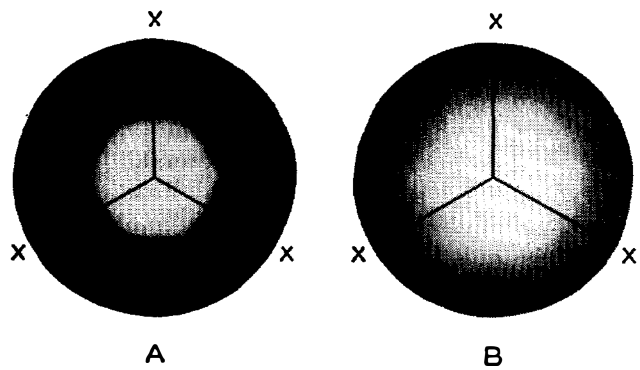

Fig. 5.20—CLEAVAGE-FIGURES may also be observed on the pin-hole oriascope in ground but unetched specimens, in this case a Z-cut section. Here the direction of the X axes but not their sense (nor handedness, nor twinning) may be determined.

bath method described above, the undistorted figure may be observed. The direction and amount of misorientation of the surface may be thus estimated. By provision of suitable mounts and scales the misorientation could be measured to 5°.

It might be added that in some cases unetched, ground (or sawn) quartz surfaces give "cleavage-figures." Thus with Z-cut sections which have been ground, but not etched, there may be observed on the pin-hole oriascope cleavage-figures like those shown in Fig. 5.20. The difference between the two views is mainly a difference in focusing and in photographic reproduction. The cleavage-figure indicates that there are preferential cleavage planes in quartz, which are parallel to the X-axes, and correspond approximately to the natural cap faces. Further, there is no indicated difference between the major and minor planes. Thus, the cleavage-figure is six-fold and may not be used to determine electrical sense or twinning. It may, however, be used to determine approximately the orientation of the X-axes. Cleavage-figures are seldom strong, but appear to be best with coarse grinding.[18]

5.6 The Process of Etching Quartz

Few factors related to the chemical process of etching quartz have been extensively studied. Much of the information here presented is taken from preliminary reports of L. Egerton of our Laboratories, who has undertaken an investigation of the etching process. Though the information mainly regards hydrofluoric acid etching, some data is given on etching with hydrofluoric gas, and bifluoride mixtures.

The reaction of quartz, which is silicon dioxide (SiO_2), with hydrofluoric acid (HF) is given by the following equations:

$$SiO_2 + 6HF \rightleftarrows SiF_4 + 2H_2O + (2HF) \rightleftarrows H_2SiF_6 + 2H_2O.$$

Since the hydrofluoric acid is a solution of HF gas in water, the reaction of the acid with quartz results in a reduction of the concentration of HF. At the same time there is produced silicon tetrafluoride (SiF_4) which reacts with more HF to give fluosilicic acid (H_2SiF_6) in solution. It is common practice to start with about 50% HF acid and to continue etching until the HF concentration is down to 20 or 25%, at which time there should also be a 30% to 35% concentration of H_2SiF_6, if all the depletion of HF were due to reaction with the quartz. Actually much smaller concentrations of H_2SiF_6 are found, and this discrepancy is mainly due to the large continuous loss of HF from the solution by gassing. Further, the etching power of this used acid is not the same as would be obtained with a solution of 20%–25% HF alone in water. However, this difference is hardly noticeable except with weak etches.

Through the useful life of the acid, starting with 50% HF and depleting to about 20% HF, practically identical etch-figures may be obtained by properly adjusting the etching time. Means of testing the etching power of the acid to determine the proper etching time are complicated by the production of H_2SiF_6 in the solution, and by the irregular loss of HF by gassing. Further, the power of the acid to produce useable etch-figures is not the same as its power to remove quartz, or to etch glass, or as its concentration of HF or H_2SiF_6. For these reasons any indirect method of measuring etching-power must be correlated empirically with the etching-time required to give the desired etch-figures.

An indirect method of testing the etching-power, developed by Dr. W. Hoff of Western Electric, Hawthorne, involves the etching of sand blasted

[18] Scrubbing the surface with soap, water, and brush sometimes improves the figure.

microscope slides for a standard length of time. The lead-glass slides become coated with a white lead-fluoride deposit to a depth dependent mainly upon the HF content of the acid. The optical density of this deposit is measured with a specially adapted photometer. The photometer readings are correlated with required etching-times to give the desired etch-figures; a different etching-time being required for different kinds of sections, slabs, etc. Use of this means of controlling the etching time has greatly improved the regularity with which good etch-figures are produced in the shop.

Commercial hydrofluoric acid from a number of different suppliers has been analyzed for purity, and tested for the development of etch-figures. It appears that when such acids are brought to the same concentration (by addition of water if necessary) there is no difference in their effectiveness, nor are they inferior to pure reagent acid. Commonly the acid is supplied as 48% solutions in lead or hard rubber drums, or as 60% in steel drums (usually the concentration is a few per cent higher than labeled). The difference in packaging is of no importance in the results obtained, provided the concentration is properly reduced.

There are two important factors regarding the starting concentration of hydrofluoric acid baths. In the first place, acids stronger than 50%, though reacting vigorously with the quartz (and removing material rapidly), do not give good etch-figures. Secondly, strong acids not contained in sealed containers lose strength very rapidly by gassing of the HF gas. Hence unused fresh acid should be kept well stoppered. Before use the acid should be diluted to a concentration of 45% to 50%. This may be accomplished by adding about $\frac{1}{3}$ volume of water to one volume of 60% acid, or $\frac{1}{6}$ volume of water to one volume of 55% acid.

Concentrated hydrofluoric acid loses HF by gassing more rapidly than it loses water by evaporation. This preferential loss of HF continues until the HF concentration is reduced to 35% or less,[19] and is not completely overcome by covering the bath without sealing. In fact, in practice, it appears that about as much HF is lost by gassing as is used in etching the quartz. Thus the bath should be kept as tightly covered as is practicable.

Whereas, in the past only lead and hard rubber have been used for fabrication of acid baths and racks, it appears that for concentrations not greater than 50% HF, copper, nickel, and brass may be used as well (steel is inferior at low concentrations). Lead-tin solders may not be used, but silver solder is satisfactory. Thus shop acid equipment may be easily fabricated out of common fabricating materials.[20]

[19] At room temperatures there appears to be a constant-concentration mixture at some concentration below the 35% concentration of the constant boiling mixture, the exact value depending upon the temperature of the solution and the ambient humidity.

[20] Polystyrene is a good material for use in fabrication of vessels for handling HF and its reaction products in the laboratory.

While agitation of the acid bath during etching does speed up the removal of quartz from the surface, it does not appear to speed up the development of the etch-figures here considered. However, moderate agitation does improve the uniformity of etch from one crystal to another, and even over the surface of single large surfaces (especially when such surfaces are close together). Uniformity of etch is important in examining for twinning. The surfaces to be etched should never be placed in contact with each other, or with other surfaces, so that the acid cannot flow between them (the separation should be at least $\frac{1}{32}$ of an inch).

The effect of temperature on the etching process appears to be small for the range of room temperatures normally encountered in practice.

A word of caution chould be added regarding the handling of hydrofluoric acid and other fluorine etching materials. The dangers are of two kinds. First, fluorine poisoning may result from contact with any fluorine compounds, the effects of which may be cumulative. Special care should be taken to prevent inhalation of vapors from all etching baths containing fluorine. Some persons are especially sensitive to fluorine poisoning. Secondly, hydrofluoric acid baths, or any baths containing free HF, may produce acid burns. Commonly such burns are attended by fluorine poisoning. For these reasons etching with all fluorine compounds is preferably carried out in ventilated hoods (with strong air suction through the door), with continually running water for washing, and with rubber gloves, tongs, racks, etc. for handling the quartz.

Etching compounds other than hydrofluoric acid have been widely used in etching glass, as is evidenced by the variety of formulae presented in the "Chemical Formulary."[21] Solutions of ammonium bifluoride (NH_4HF_2), with additions of various amounts of free hydrogen fluoride, sodium bifluoride, sugar, and other materials have long been used on glass. One of the possible advantages of such formulae for etching quartz is the elimination of the dangers of acid burns and strong fumes that may be obtained with hydrofluoric acid (care must still be maintained to prevent fluorine poisoning). A number of these formulae have been made up and tested on quartz. The preliminary conclusions are as follows.

The etch-figures that may be developed by the bifluoride compounds on Z and X-cut sections of quartz are not the same as those developed by hydrofluoric acid. The results approach each other, however, for excessively long etching in both cases. To obtain *usable* etch-figures on X-cut sections with the bifluoride requires considerably longer etching time than with hydrofluoric acid, or an elevation of the bath temperature to about 45°C. The addition of hydrofluoric acid to the bifluoride formulae speeds up the development, but partly negates the safety advantage of the bifluoride bath.

[21] Published by the Chemical Publishing Co., Brooklyn, N. Y.

The figures produced on Z-cut surfaces are small and complex, (hardly usable) unless a considerable amount of free HF acid is added. Etch-figures here considered are those produced on the pin-hole instrument, and are usable only if they have such character as will permit of their use in determining quartz axes. Fig. 5.21 shows the type of usable etch-figure obtained on X-cut sections with an ammonium bifluoride and sugar solution (the sugar is here effective mainly in preventing creepage of the solution). It might

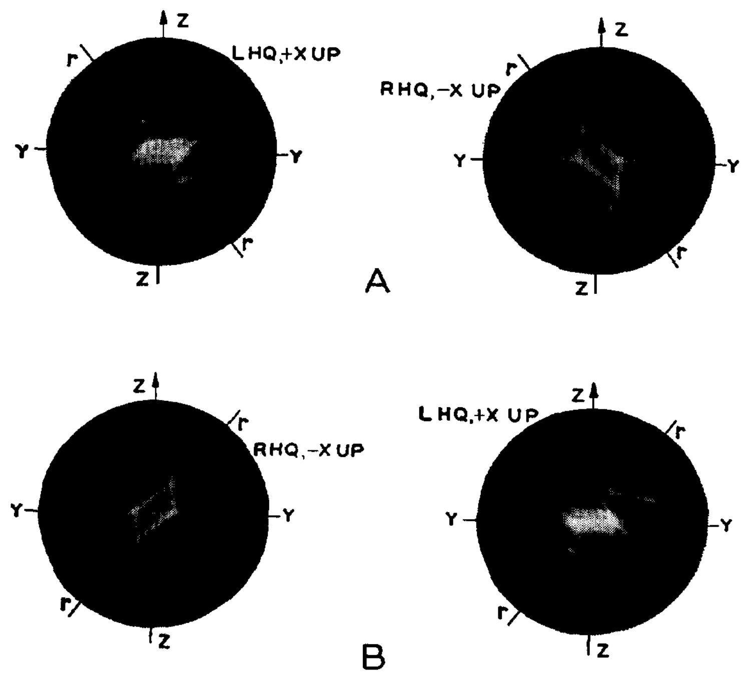

Fig. 5.21—Etch-figures obtained on the pin-hole oriascope with X-cut sections which have been strongly etched in bifluoride mixtures, or excessively etched in hydrofluoric acid. These etch-figures differ from those of Fig. 5.19 (for a moderate etch in hydrofluoric acid) but are obviously usable.

be noted that a similar figure is obtained with hydrogen fluoride gas, and with *excessively* long etching (several hours) in hydrofluoric acid.

When the bifluorides are used only to develop reflection contrast in the detection of twinning, their effectiveness appears to be about the same as hydrofluoric acid, under equivalent process conditions. The etching power of the bifluorides may be maintained nearly constant over a long period of use by maintaining an excess of the salt in solution, a distinct advantage over the acid. The metals copper, nickel, brass and stainless steel may be used in fabricating tanks and racks, lead and steel are inferior.

Finished quartz surfaces are sometimes etched to remove surface debris (fragments of quartz loosened by grinding, and grinding refuse embedded in microscopic surface irregularities), and to remove predetermined small amounts of the surface for frequency adjustment. It is common for these purposes to use weaker etching solutions, since very small amounts of quartz are to be removed. With hydrofluoric acid, weak solutions (less than 20% HF) have an advantage in that their concentrations are little reduced by exposure to the air. In fact with very weak solution the concentration may increase slightly by exposure, and thus partly compensate for the HF lost by reaction. Weak ammonium bifluoride solutions may also be used, provided no deposit forming material is added.

5.7 The Effect of Twinning in the Finished Plate

While it is commonly considered that electrical and optical twinning are not allowable in a finished oscillator plate, it cannot be unconditionally stated that small amounts of twinning will too seriously affect the properties of all types of oscillator plates. The allowance of even small amounts of twinning in the finished plate would save quartz and simplify the processing procedures. Hence, consideration must be given to the factors which would affect the utilization of twinned material, and the effect of twinning on the operating characteristics of the finished plate. Consideration will first be given to the nature and distribution of electrical and optical twins[22] in the raw quartz.

The analysis of twinning in raw quartz has been carried out by the examination of numerous, etched Z-cut surfaces. By the method to be described it is possible to detect the handedness, and the axial orientation and sense, of each homogeneous portion, twin, appearing at the etched surface of a twinned specimen. Both electrical and optical twins may be analyzed by this method. It might be added that electrical twinning boundaries and orientation are only detectable at an etched surface, and that while interior optical twinning may be detected by polarized light, its exact analysis is only possible at an etched surface.

Figure 5.22 E shows the optical arrangement used for examining twinning in etched Z-cut sections. The sections (prepared with a fine grind and weak etch) were mounted on a turntable, illuminated from an elevation of about 30° to the horizontal etched surface by a spot lamp, and viewed (or photographed) from vertically above the section according to principles of Section 5.41). With the section properly aligned on the table (with the predetermined electric axes parallel to the table-lines joining diametrically opposite fiducial marks), the table was successively turned into positions about 12° to the right or left of the plane of illumination and reflection (as indicated by the R and L marks and the index pointer). Four of these positions of

[22] See footnote 4.

illumination of a given section are sufficient to determine the nature of the four possible twins in the section. The four corresponding photographic views of the section have been arranged in a special manner to simplify their explanation. This arrangement, as shown in Fig. 5.22A, B, C and D, is equivalent to what would be observed if one looked down on a single, stationary section, and illuminated the section from the four different direc-

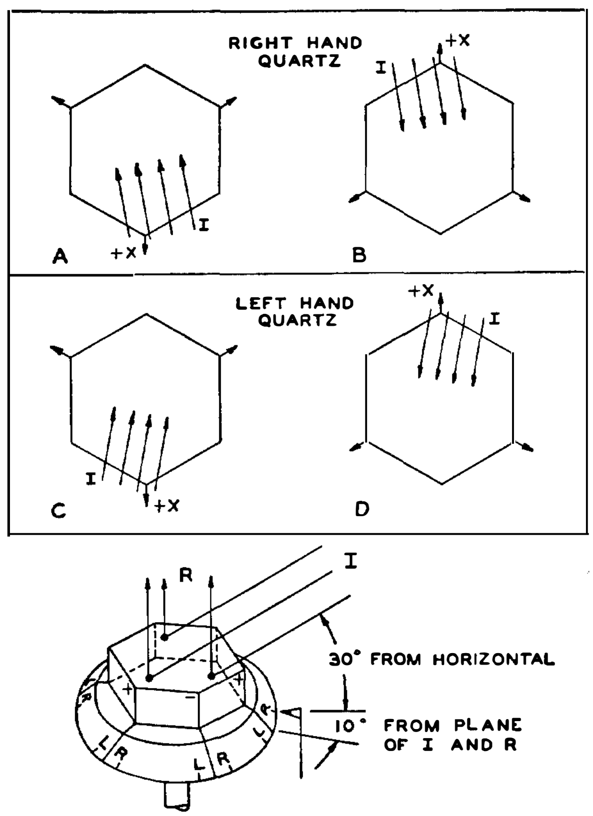

Fig. 5.22—Reflection patterns of the twinned, Z-cut sections shown in Figs. 5.23, 5.24, 5.25 and 5.26 were obtained by the means shown in E. A, B, C, and D are a key to the four equivalent directions of illumination of a single stationary section.

tions shown in the figure. For each direction of illumination there is a corresponding view, the outline of the section (and any cracks, chips or other flaws) being identically positioned in each view. However, when the four types of twins are present in a given section, each view will show a different region, or regions, of brightness. For each view, the interpretation of handedness and electrical sense of the *bright portion* of the view is according to the labeling of this particular view, only. Thus if a section is entirely right quartz and of the electrical sense shown at A the whole surface of the section will appear bright in view A and dark in all other views. If a section is all right quartz, but partly of the electrical sense shown in A and partly that shown in B, then part of the surface will appear bright in A and the other part will be bright in B (the whole surface will be dark in C and D.)

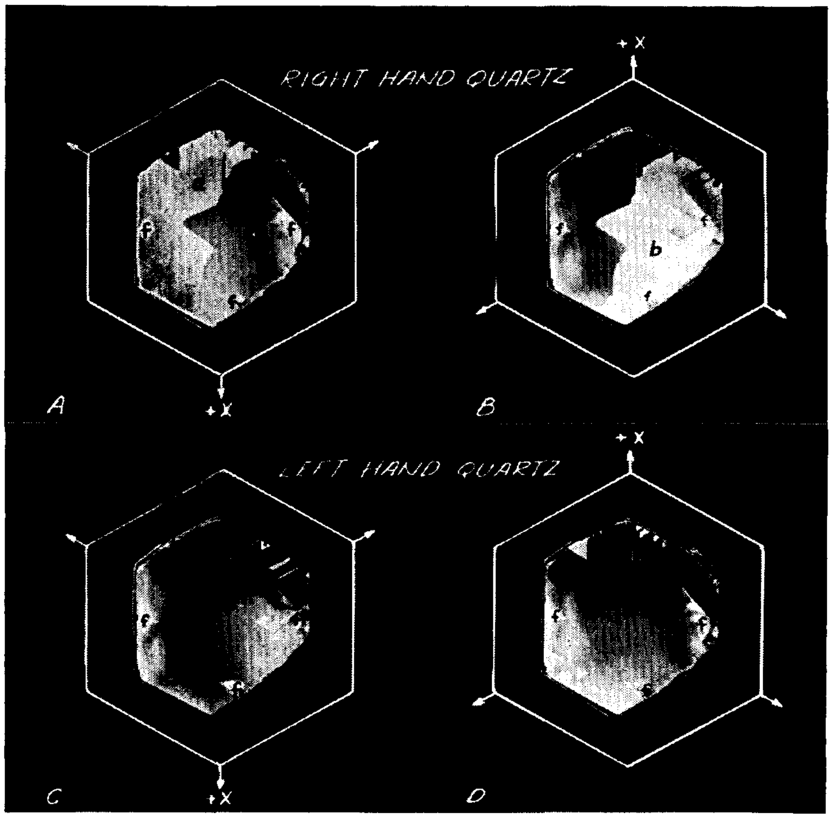

Fig. 5.23—The four possible conditions of handedness and electrical sense in a single section are shown here. In each view the handedness and sense is for only the ***bright*** portion of that view. The *a* and *b* regions are seen to be both of right quartz but of opposite electrical sense, hence electrical twins. (Flaws indicated by *f* are to be disregarded).

A section containing all four possible twins would exhibit bright regions in each view, and a different bright region in each view. All bright regions would fit together to make a complete map of the surface. Only the bright portion of each view has the handedness and electrical sense indicated for that view.

Figure 5.23 shows a Z-cut section containing twins of the four possible conditions of electrical sense and handedness. The two large, bright regions *a* and *b* (appearing in views A and B respectively) are both right quartz but of opposite electrical-sense. Hence the surface is mainly of electrically twinned right quartz. The small dark regions within the borders of *a* (view A) are bright in view D. Hence these small, triangular and line regions are left quartz and of opposite electrical sense to the large region *a* containing them. They are then optical twins of the large *a* region. Similarly the dark regions of *b* (view B) are found from view C to be optical twins of the *b* region. (Flaws labeled *f* are cracks, chips, etc.) If the whole section were cut up to make AT plates, for example, and at the proper angular sense according to the *a* portion of the section, then those plates coming from the *b* region would be of wrong angular sense. Those crossing a boundary between the *a* and *b* regions would be of both senses, i.e., electrically twinned. Those few plates which contained some left quartz would be optically twinned. To make the most economical use of this section it should be separated, by cutting along a line approximating the *a* to *b* boundary, so that each half of the section may be cut at the correct sense of orientation. Even when so cut, some of the plates will contain optical twinning and remnants of electrical twinning. This section is typical of much of the raw quartz that must be used for manufacturing piezoelectric plates.

Figure 5.24 shows a section which is mainly of left quartz as exhibited by the large bright *c* and *d* regions of views C and D. The large *c* region is optically-twinned to a small extent by the line regions *b* of view B. One of the *d* regions is badly optically twinned by the small striated *a* regions, as seen in A. Such a section would be very uneconomical to process, since separating the larger electrical twins is not feasible. If processed at all, it should probably be entirely cut according to the handedness and sense of the large *c* portion, the wrong-sensed regions and twinning being cut away at a later stage (after inspection of the slabs in the twinoriascope, for example). It might be noted that only the optical twinning could have been observed in the initial polarized-light, raw quartz inspection, where such a stone would be passed as moderately good.

Fig. 5.25 shows an ***unusual*** section that is mainly composed of left quartz, regions *c* and *d*. The right quartz regions shown in view B are of both opposite-handedness and electrical-sense to the *c* region inclosing or bordering them. This is the common and expected conditions. The unusual condition is exhibited by the regions *c* and *a*, where twins of opposite-handedness but ***same*** electrical-sense have a common boundary. Since this boundary could be detected by optical means, the *a* and *c* regions might be described as optical-twins, of an "uncommon variety". However, by convention ***optical twinning*** has long been used to denote twinning exhibiting both opposite-handedness and opposite-electrical-sense (crystallographically, Brazil twinning). Further, twinning exhibiting both opposite-handedness and same-electrical-sense, combines the crystallographic twinning laws of Brazil twinning and Dauphiné (electrical) twinning. Hence this uncommon variety of twinning may preferably be called combined electrical and optical twinning, or just COMBINED TWINNING. Thus, the boundary

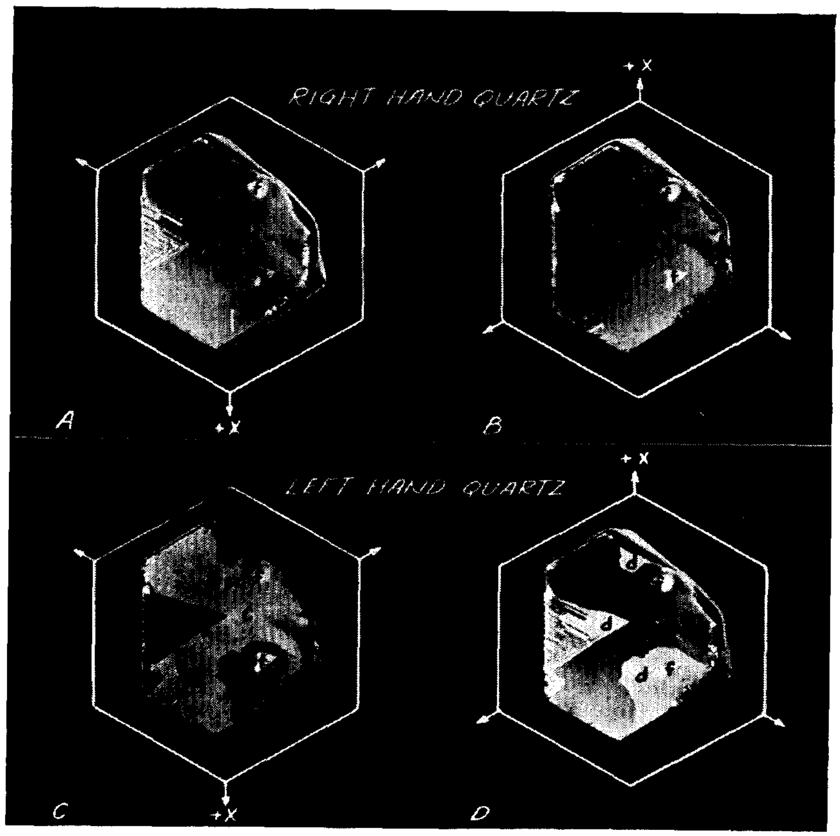

Fig. 5.24—Regions *d* are electrical twins of the region *c*. The striated regions *a* are of opposite handedness and electrical sense to the *d* region enclosing them, hence optical twins of *d*. The *b* regions are small optical twins of *c*, and *f* are flaws.

between the *a* and *c* twins separates combined twins. Note also that the *a* twin bounds the *b* twin and the *b* twin bounds the *c* twin. Thus, *a* and *b* are true electrical twins, and *b* and *c* are true optical twins.[23]

[23] It is possible that growth conditions are such that combined twinning cannot occur by itself, without the presence of true optical twinning and true electrical twinning. That is, a region of given handedness and sense can not be entirely bordered by a region of opposite-handedness and same-sense.

Figure 5.26 shows an unusual section which is mainly composed of left quartz, of the electrical sense shown in D, region *d*. The region *c* is an electrical twin of *d*. The region *f* is a flaw in the quartz and is to be disregarded. The region *a* is an optical twin of *d*, and is *uncommonly* large for an optical twin (note: region *a* contains within it, two small optical twins). Since

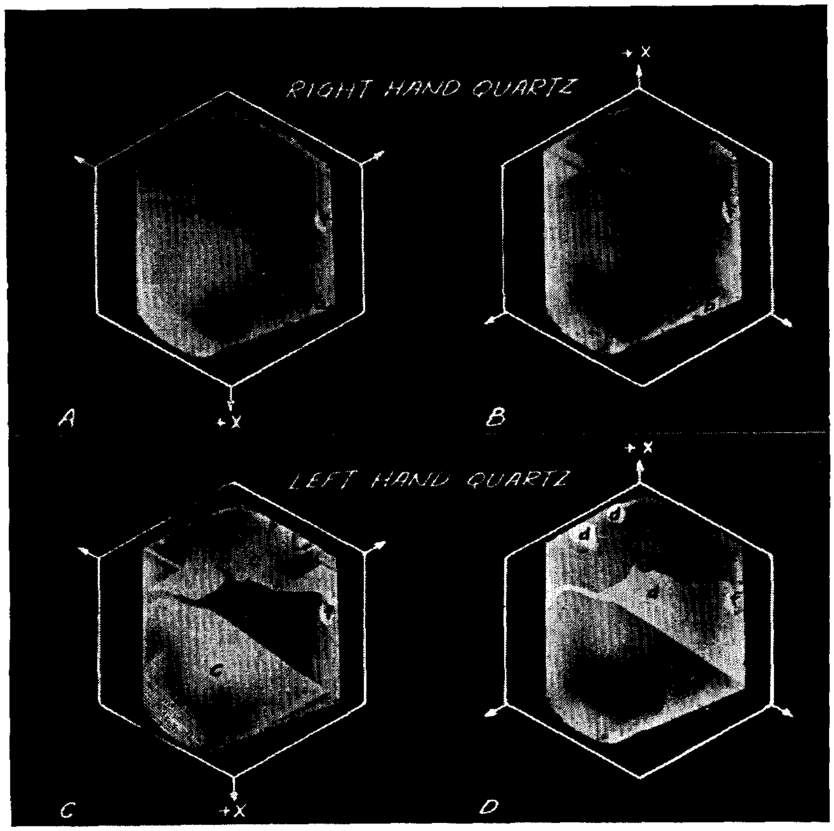

Fig. 5.25—Regions *c* are electrical twins of the adjacent *d* regions, *a* is an electrical twin of *b*, and *a* is also an optical twin of *d*. An uncommon condition of twinning is presented by the adjacent *a* and *c* regions which are of opposite handedness but the *same* electrical sense, thus exhibiting COMBINED-TWINNING.

optical twins are usually very small (except for the one major surrounding twin), it is seldom possible to cut them apart and use each twin individually.

Figures 5.1, 5.2 and 5.3 were obtained by the means above described, and all sections shown in these figures (except Fig. 5.2A and C) actually exhibited both electrical and optical twinning. Thus Fig. 5.3D was obtained from Fig. 5.24A, and Fig. 5.2F from Fig. 5.24C, etc., by trimming the latter named figures to give the sections simulated natural faces. Figures 5.2 and 5.3 are of particular use in learning to distinguish between electrical and optical twinning when examining etched surfaces by reflection. Note that electrical twins are usually large and separated by irregular boundaries, Fig. 5.2. Optical twins are usually separated by straight-line boundaries

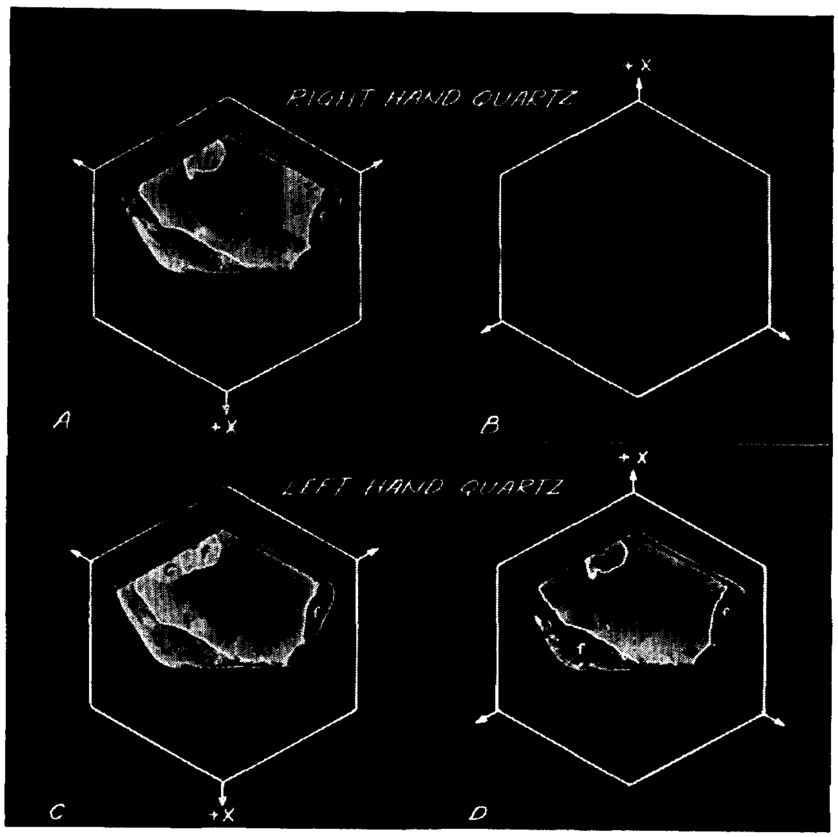

Fig. 5.26—Since this section exhibited no bright regions (except flaws *f*) in view B (i.e. no right quartz of electrical sense B) it was not reproduced in view B. The *c* region is an electrical twin of the adjacent *d* region, while *a* is an optical twin of *d*. It is *uncommon* for a minor optical twin to be as large as *a*.

parallel to natural faces, thus forming triangular, parallelogram, and straight line insets, Fig. 5.3. Optical twins (except for the one major, surrounding twin) are usually very small and often interlayered (with the major twin). Large interlayered regions are entirely unusable and hence are cut away at the earliest possible stage to save the labor of processing worthless material.

Small optical twins and small electrical twins (or remnants of electrical twins left after cutting electrical twins apart) may be isolated or removed in an intermediate or late stage of processing, where they are detected by the etch technique. Commonly the final rejection of material twinned in either way is delayed until after the final blanks are cut out. These may be etched and examined by reflection, one at a time under a spot lamp, and those showing twinning (and other imperfections) sorted out and rejected.

Another possible method of rejecting twinning which is of sufficient amount to be harmful is by making electrical tests on the finished (or semi-finished) plates, at which time those plates failing to meet the electrical tests for any reason (including twinning), are rejected. While this method of rejection does not assure that twinning will be entirely absent from the accepted plates, neither does any other method assure complete absence of twinning. Further, except for imperfections which may affect the useful life of the plate, acceptance of finished oscillator plates is not illogically based

TABLE I.—*Constants for Plates of Correct and Incorrect Sense of Cut*

Cut, Angle	Frequency Constant (fxd. in Kc. 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)

upon their meeting the desired electrical operating characteristics, i.e., frequency, temperature-coefficient, activity and internal damping (all determinable by electrical tests).[24] It does not appear that twinning will affect the useful life of the plate. Its effect upon the electrical operating characteristics of the plate depend upon many factors.

An important factor regarding twinning in the finished plate is that optical twinning introduces a less important variation in the physical properties of the plate than does electrical twinning. Thus, in the case of optical twinning alone, both portions of the plate are of the same sense of cut, though still being of opposite electrical sense. This may be understood from an examination of Fig. 5.4, the second and third views taken together represent optical twinning. In the case of electrical twinning the two portions of the

[24] With filter plates additional operating characteristics must be met. The ratio of capacities (see Chap. I, Appendix A.3) is greatly affected by the opposed electrical sense of twinning.

plate are of both opposite sense of cut and opposite electrical sense, as may be observed from the third and fourth views of Fig. 5.4. The effect of this difference in sense of cut for the two types of twinning is brought out by Table I, which gives the approximate frequency constants and temperature coefficients for the common cuts of oscillator plates, together with those for the analogous, oppositely (and hence wrong) sensed cuts.

In the case of a CT plate, for example, both portions of an *optically twinned* plate (cut at $+38°$) will be of the same $+38°$ orientation. The plate is elastically the same throughout and hence should exhibit the frequency and low temperature-coefficient desired. However, the opposed electrical senses of the two portions will cause a reduction in the electrical activity. The amount of this reduction will depend upon the relative size of the two portions and upon their placement relative to the vibration nodes of the plate.

On the other hand, when a CT plate is *electrically twinned* one portion of the plate will be of the correct $+38°$ orientation while the other portion is of the incorrect $-38°$ orientation. The two portions of the plate have widely different elastic properties, as is exhibited in the table by the different frequency constants and their respective temperature-coefficients. Resulting from this difference alone, the plate will exhibit operating characteristics (if operable at all) intermediate between the two listed in the table (usually near one of these two), and its activity will be reduced. The activity will also be reduced by the opposite electrical senses in the two portions. The degree to which the frequency, temperature-coefficient, and activity are affected, again depends upon the relative sizes of the two portions of the plate and their placement relative to the "nodes" of the plate.

Thus, for equivalent proportions and placement of twinning, electrical twinning will cause a much greater change in the operating characteristics of the plate than will optical twinning.[25]

A note may be inserted regarding the electrical testing of plates, some of which may be twinned while others may be untwinned but of incorrect sense of cut. As seen from Table I, untwinned plates of the correct sense of cut are easily distinguished from those of the incorrect sense of cut by their frequency. This distinction between sense of cut holds as well for plates containing very little twinning. The presence of appreciable twinning in the plate is easily distinguished by the activity of the plate. While ordinarily a plate would be electrically tested in the mode of vibration it is intended to be operated in, it is sometimes of advantage to test it in a different mode.

[25] In the case of the *uncommon* "combined-twinning" the two portions of the plate are of opposite sense of cut but of the same electrical sense. The effect on the operating characteristics will be like that for electrical twinning, except that the activity may not be as greatly reduced.

Thus the high-frequency mode plates (AT and BT) might be tested in their low frequency modes (corresponding roughly to the CT and DT modes, respectively). An electrical crystal-sorter using these principles has been developed by I. E. Fair, see Fig. 16.40.

5.8 Conclusions

In the processing of quartz, consideration must be given to the nature of twinning and to its characteristic distribution in the raw stone. There are only two common types of twinning that need be considered, namely *electrical twinning* and (true) *optical twinning* ("combined-twinning" and other types are a rarity). Due to the characteristically large size (and the nature) of *electrical twins,* a stone must be examined for electrical twinning (by the etch technique) at an early stage of processing so that the electrical twins may be observed and cut apart before the angular cuts (AT, BT, CT, DT, etc. slabs, bars, or wafers) are made. Otherwise, some of the large electrical twins will be entirely cut up with the incorrect angular sense, and hence wasted.

On the other hand *optical twins* are characteristically small and interlayered, or small and scattered. The interlayered regions are entirely unusable. Hence processing labor will be saved by inspection of the raw stones (by the polarized light means of Chapter IV), and of the first sections at least (by the etch technique) for large regions of interlayered optical twinning.

Scattered optical twins and small electrical twins, or remnants of electrical twins which have been cut apart, may be cut away in an intermediate processing stage, or in a later stage plates containing such twinning may be separated out. In either case the etch technique may be used to detect the twinning.

An alternative method of eliminating small electrical twins (or remanents thereof) and of small optical twins (most of which are characteristically very small) is by electrical tests on the finished plate. This method has merit in that if the twins are sufficiently small, and not disadvantageously placed in plate, they may not harmfully effect the desired operating characteristics of the plates. The degree of the effect depends not only upon the size of the twin and its location in the plate, but upon whether the twinning is electrical or optical; *optical twinning* being considerably *less harmful* than electrical twinning. The effect of the twinning further depends upon the type of plate being considered, i.e. its size and mode of operation, and use. It is probable that twinning is more tolerable in low-frequency mode oscillators (CT and DT) than in the high frequency modes (AT and BT), and of course more tolerable in plates of low requirements on the operating characteristics (activity, frequency and temperature-coefficient). Twinning is probably least tolerable in filter plates, which have to meet very special requirements.[26] Detailed experimental studies of allowable amounts of twinning are of little value since to use the results in a manufacturing process would require a careful inspection of each plate and a difficult classification into groups depending upon the variety, amount, and placement of the twinning. Acceptance or rejection of finished plates on the basis of their final electrical operating characteristics appears to be the only practical means of separating usably twinned plates from unusably twinned plates. This method of selection does not determine whether the rejected plates contain twinning or other imperfections (or are misoriented or misdimensioned) and is therefore of little use in analyzing the processing methods to determine best practices. This disadvantage may be eliminated by etching the *rejected* plates and examining them for twinning (and such other imperfections as show up best after etching).

The effects of crystal imperfections other than twinning were discussed in Chapter IV, Section 4.9.

[26] See footnote 24.