CHAPTER IV

Raw Quartz, Its Imperfections and Inspection

By G. W. WILLARD

4.1 Introduction

QUARTZ is one of the commonest of crystalline minerals and occurs in many variations of size, color, purity, and structural perfection. It is used for such varied purposes as jewelry, fusing into heat and chemical resistant dishes, and for optical and piezoelectric units. However, the following discussion will be concerned mainly with such raw quartz as is commercially used in the manufacture of piezoelectric circuit elements. It might be added that the terminology used may be more in keeping with the language of the piezoelectric manufacturers than of the geologist. Further, description of many unusual types of defects, and variations of common types has been omitted. An attempt is here made to describe such defects as are of most interest in the piezoelectric art and in such a manner as to be most widely useful. Following is a description of raw quartz and its defects, the means of observing these defects, their appearance as recorded photographically, and a discussion of their effects on finished plates.

The words *defects* and *imperfections* as used in this article mean a deviation from a perfect specimen of raw quartz; they do not necessarily mean that the material is not entirely satisfactory for the purposes intended.

4.2 Source, Size, Shape

Quartz crystals of usable quality and size come mainly from the interior of Brazil. From other sources the supply is negligible, or the size too small, or the imperfections too predominant. Even from Brazil only one in a hundred of the mined stones is usable. The size of stones most commonly used run from one-half to five pounds (about one-half cup to one quart size). The shape of the stones varies from well faced material, with all of the original natural faces intact, to stones in which the faces are broken or eroded away. When the faces are entirely broken away by mining operations the stones resemble chunks of broken glass. When the faces are eroded away by having been washed along river beds the stones are called RIVER QUARTZ, and the appearance is that common to river stones. River quartz usually exhibits a network of shallow surface cracks resulting from the continual bumping along a river bed, and hence is more subject to thermal and mechanical shock than uncracked stones.

With defaced quartz (river and broken) the orientation of the crystal structure cannot be determined from the surface shape. Since the stones must be cut at specific orientations relative to this structure, special means must be employed to determine the structure orientation. For this reason many users of quartz prefer faced stones. However, defaced stones are usually more free from defects than faced stones, since optical twinning is commonly concentrated near the natural faces of the original stone and other defects near the base. Thus by making use of special means (inspectoscope, conoscope, oriascope) defaced quartz may be cut to good advantage.

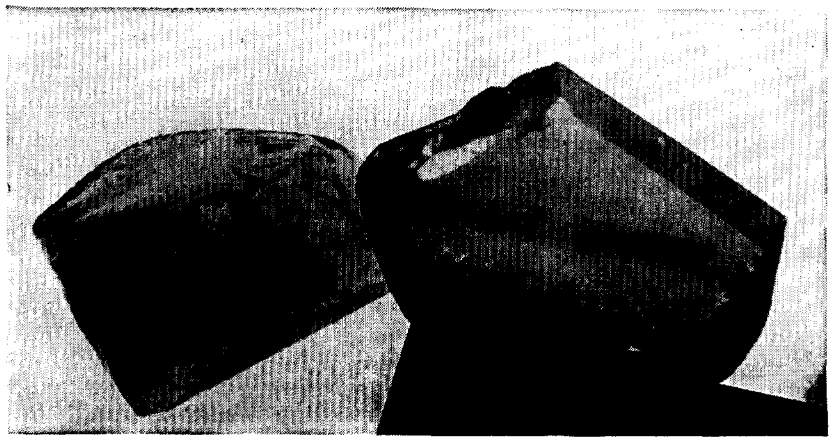

Fig. 4.1—Quartz may show smoky or citrine coloring throughout or only in restricted regions, irregularly as at the left, or in PHANTOM planes as at the right.

4.3 Color

Usable quartz is transparent internally (though the exterior surface may be opaquely coated), never translucent (milky). The color of the quartz varies from perfectly clear through slightly smoky to fairly dark. The obviously dark stones are called SMOKY QUARTZ. Smokiness may be uniform throughout a piece, or varying from clear to dark, or confined to plane sheets within a single piece, see Figure 4.1. Dark smoky stones are not used because they cannot be inspected for defects and optic axis. With stones that are used this coloration is seldom so dense that it may be detected in the small finished plates with ground surfaces.

Because the smokiness is due to so slight a deviation from the pure quartz its analysis is extremely difficult. The coloration is variously explained, as due to minute traces of impurities (organic or inorganic), as due to the dissociation of a few SiO_2 molecules into free silicon and oxygen, and otherwise.

An important fact about smoky quartz is that it may be cleared of coloration merely by heating to 350°C. to 450°C. for a short time[1] (see Fig. 4.2). The clarity of cleared stones, even though originally very dark, rivals that of normally clear quartz, thus leading to the belief that most commercial quartz is colored to a slight degree.[2] Further, it is known that irradiation of either cleared or normally clear stones with X-rays may cause them to become

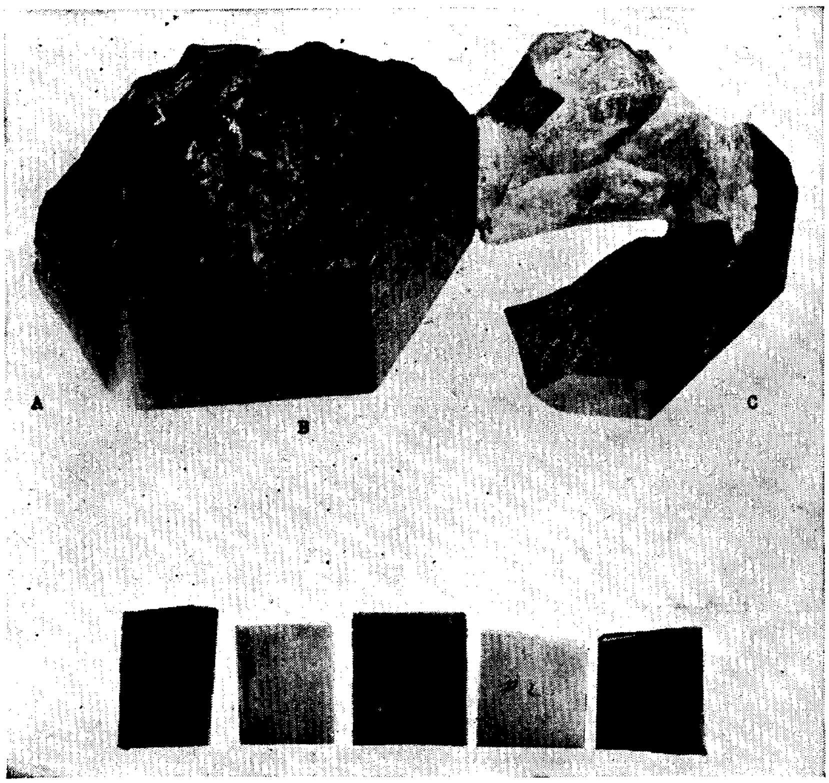

Fig. 4.2—Coloration in quartz may be cleared by heating to temperatures below 500°C. A, B and C are adjacent portions of one dark, smoky stone, only C having been cleared. The five, small blocks were likewise cut from a single stone, the second and fourth having been partially cleared at 350°C. and fully cleared at 500°C.

smoky. It is then questionable whether there is a material difference in clear and smoky quartz, or only a difference in condition of the material. By the limited studies that have been made light smokiness has not been found to have any definite effect on finished plates. It might be added that such quartz plates as are normally heated to 400°C. to 500°C. in the process of manufacture, would of course be cleared of any smokiness originally present.

[1] Stones so cleared 8 years ago are still clear.
[2] Arkansas quartz (not used commercially) is noted for its unusual clarity.

What has been said of smoky quartz applies equally well to CITRINE QUARTZ except that here the coloration is straw colored (yellow to brown). Citrine quartz is even more uncommon, commercially, than smoky quartz.

4.4 Twinning

Electrical and optical twinning are among the most common defects of crystal quartz. There are few stones without one or both present. Either type is usually difficult or impossible to detect from the exterior form of the crystal. Presence of either type in a finished piezoelectric element interferes with its perfect operation. Twinning is an abnormality of growth, in which an apparently homogeneous crystal is not actually of the same handedness, electrical sense, or orientation throughout. In the case of both electrical and optical twinning (the only common twinning types in quartz), the electric and optic axes in all parts remain parallel each to each.

In a crystal which is only ELECTRICALLY TWINNED, the stone is entirely of one handedness (either right or left), but one portion is of OPPOSITE ELECTRICAL SENSE to another portion. This change in sense of structure is NOT detectable by ordinary optical means. However, at the surface it is detectable by, (1) the piezoelectric effect (determination of electric charge on squeezing); (2) x-ray reflection intensities (using certain sense determining planes, see Chapter III); and (3) most readily and extensively, by etch-pits and etch-pit figure techniques.[3] Commonly electrical twins are sufficiently large that they may be separated near a twinning boundary and both parts used.

In a crystal which is only OPTICALLY TWINNED, one portion of the crystal is of OPPOSITE HANDEDNESS and electrical sense to another portion. This change in handedness of structure is detectable by optical means (i.e. by examining between crossed polarizing filters). Optical twinning may also be detected, at the surface, by the etch technique in the same manner as electrical twinning. Usually a stone will be mainly of one handedness with only small, thin, interlayered growths of opposite handedness, thus making it impossible to use both handed portions separately.

Further discussion of both electrical and optical twinning will be found in Chapter V, where means for simultaneously detecting both are described.

4.5 Cracks

Many quartz stones contain cracks which are not readily seen by a casual surface examination. As mentioned above river quartz commonly exhibits a network of shallow cracks extending inward from the surface, caused by bumping. All types of stones commonly contain one or more cracks, es-

[3] In finished plates, of course, the effects of twinning are also determined by measuring the resulting piezoelectric and elastic constants of the plate (i.e., their effect on frequency and activity).

pecially when badly twinned or full of inclusions. Some of these cracks may be due to rough handling, but others are due to growth conditions, or result from temperature changes after growth.

Though large cracks are readily detected by means to be described, it might be noted that when cracks are sufficiently fine (small separation compared to light wave-lengths) they will no longer be seen. Thus every visible crack may be considered to extend beyond its visible range, and some actual cracks will not be visible at all. Because of such cracks and other defects in quartz, special care should be taken in handling uncut and partially cut stones to prevent their subjection to mechanical or thermal shocks. On the other hand, small, flawless, finished plates will stand considerable shock. It is even common practice to solder to finished plates (after metallizing).

4.6 Inclusions (Bubbles, Needles, Phantoms, Veils, Etc.)

The remaining abnormalities of raw quartz (used for piezoelectric elements) may be classed as inclusions. Among these are inclusions of solid, liquid, and gaseous material. The size of individual inclusions may vary from submicroscopic, to those easily visible with the naked eye. The inclusions may be isolated, or arranged in lines, or planes, or curved surfaces. In many cases the arrangement forms of inclusions (bubbles, needles, phantoms, veils) have been used to describe inclusions, with little regard to the nature or size of the individual inclusions. This is because inclusions which are too small to analyze individually, are still visible when grouped by hundreds in lines or surfaces.

When inclusions are sufficiently small and closely grouped they give a BLUISH cast (Tyndall effect) to the group. Thus the bluish cast is recognized as indicating fineness of grouped inclusions. When the individual inclusions are larger, the group appears white. With still larger inclusions one may actually see separate, individual inclusions, looking like minute bubbles. Thus, describing the group as blue, white or bubble textured is of considerable importance, when analyzing or estimating the usability of quartz with grouped inclusions.

BUBBLE INCLUSIONS look like small bubbles (i.e., small spheroidal cavities) in the quartz. When bubbles appear individually, or randomly scattered, they are referred to as just bubbles. When bubbles occur in organized groups the group is referred to as a bubble phantom, bubble veil, etc. Smaller bubbles appear only as light reflection points and their shape is not seen.

In general such bubbles may be filled with gas, liquid, solid, or any combination of phases. They may be of the same nature as rare, large cavities in which one can easily see a liquid moving about. Analysis of the contents of such cavities has indicated the presence of CO_2, water, salt solutions, and other substances that might have been present during growth conditions. That these cavities are seldom in the form of negative crystals, i.e. having the plane natural faces characteristic of quartz, is not easily explained.

NEEDLE INCLUSIONS appear as long, thin lines or needles. They may be straight or curved, blue, white or otherwise. Whether they are continuous or composed of rows of individual inclusions is usually not discernable. Needles, visible without concentrated illumination (and fluid immersion), are likely to be inclusions of crystalline material, such as rutile (brown), tourmaline (black). Usually such needles are called rutile needles, because of the commonness of rutile and the difficulty of determining whether they are rutile or some other material. They might better be called DARK needles. BLUE NEEDLES are fine textured, and may appear singly or in parallel groups, which may be at angles to other parallel groups. In other cases they spread from a bubble point, like a comet. Blue needles may also be feathered (having short feathery rays along the sides), may be hard (very fine and sharply distinguishable), or soft (diffuse). Probably the most important characterization of all blue needles is their blueness, which indicates fine texture. WHITE NEEDLES are similarly hard or soft. CHUVA is a special type of white needle which would be extremely elusive except for the fact that along its length are small bubbles, giving chuva the appearance of dew drops along a thin fiber. For piezoelectric usage an important distinction between needles is whether they are blue, white, chuva, or dark.

PHANTOMS are an arrangement form of inclusions (or coloration), in plane sheets which are parallel to possible natural crystal faces (usually the prism or pyramid faces). Often several, differently oriented, phantom planes are formed together so as to give the appearance of a crystal within a crystal, thus the name phantom (or ghost). Phantoms may also appear as groups of parallel sheets. Phantoms may be of smoky, blue, white, or bubble texture and should be so noted when describing their effects on piezoelectric elements. That phantoms are closely related to disturbed growth conditions is apparent from their close relationship to crystal faces.

VEILS are an arrangement form of inclusions in curved sheets. They are most commonly of a tenuous bubble texture, but may also be white or bluish. Again this distinction is of importance in estimating their deleterious effects. The cause of inclusions appearing in veil form is not clear.

CLOUDS (a term not widely used) refer to inclusions irregularly distributed in restricted regions of the crystal.

4.7 Inspection Means

The raw quartz inspectoscope is the name of an instrument used for the inspection of raw quartz. This inspectoscope may, of course, be also used for the inspection of quartz in various stages of processing, and for other transparent materials than quartz.

By means of a polarized light optical system the stones are examined for optical (but not electrical) twinning. By this same means the direction of the optic axis through the stone is also determined. By means of concentrated high-power illumination the stones are inspected for cracks, color and inclusions. By both means the stones are illuminated and inspected while immersed in an immersion fluid of matching index of refraction.

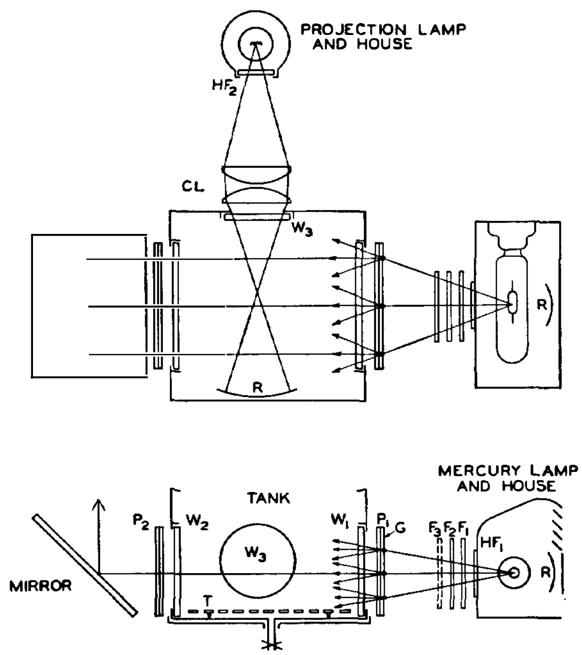

Fig. 4.3—The raw quartz INSPECTOSCOPE, top and front views. The stones are held in the immersion tank and viewed from above. Polarized light from the mercury lamp is used for locating the direction of the optic axis and for detecting optical twinning. Concentrated light from the projection lamp is used for detecting other interior defects. See also Fig. 4.5.

Without such immersion it is difficult or impossible to illuminate the interior properly, or to see into the interior, of oddly shaped or rough surfaced stones. Such immersion eliminates refraction (bending of light rays) at the surface. The interior of stones with ground, fractured, or other surfaces, are as easily examined as a piece of plate glass.

The important design features, maintenance and operation procedures are given below. Figure 4.3 is a diagram of the instrument, and Fig. 4.5 shows an early model utilizing many of the features described below. Three plate glass windows W are cemented into apertures in the open-top, steel immersion tank, Fig. 4.3. A raised perforated table T rests on the bottom of the tank. For polarized light inspection of twinning and of optic axis direction, the right to left optical system is used. This comprises an AH-4, 100 watt, mercury-vapor lamp (requiring special transformer) isolated in a well ventilated housing with window HF_1, a set of color filters F_1, F_2, (and possibly F_3), a polarizing filter P_1 with ground surface G (for light diffusion), the immersion tank, the polarizing filter P_2 (crossed to P_1), and the mirror (mounted at 45° to the vertical) to reflect light vertically up through a window in the drain pan (not shown in Fig. 4.3), to the eye. For inspection of defects other than twinning, a high powered projection system is used. This comprises a projection lamp (isolated in a housing by window HF_2, and with forced draft), condenser lenses CL, and the immersion tank. Here one looks down directly into the tank at the stone. Reflectors R may be added to both systems to increase the illumination.

This instrument resulted from a restudy of long-used "inspection tanks" and methods, and includes some features not originated by the author. Since this inspectoscope is believed to be superior to many inspection-tank equipments now in use, the more important design features will be described.

The tank should be large enough to allow easy handling of the stones in the fluid, with allowance for positioning any portion of the stone in the projection beam, and allowance for rise in level of the fluid as the stone is immersed. However, the size should not be made larger than necessary, for it has been found in practice that the fluid very rapidly collects lint and dirt, which (being kept in suspension by agitation) scatters or diffuses the light. This, besides scattering the light from its proper paths, interferes with the polarized light inspection by depolarizing the beam. In large tanks with dirty oil the light becomes almost completely depolarized and no patterns can be seen. If, however, the crystal is large enough to nearly fill the length of the tank (along the polarized light beam), this depolarization is small.[4] Thus, a tank as small as is consistent with the size of the stones to be examined should be used. The smallest convenient size and shape of tank would be about 8 inches in the polarized light direction, 8 inches in the projection light direction, and $5\frac{1}{2}$ inches high (all elements of Fig. 4.3 are drawn in proportion to these tank dimensions). This permits easy examination of two to three pound stones (pintsized), and six-inch long stones may be used without great difficulty.

Isolation of lamp heat is an important consideration in both optical systems. In the projection system the high wattage lamp would dangerously heat up the whole instrument if the heat were not properly dissipated. In

[4] In an emergency small stones can be examined in a large tank with polluted fluid by placing the stone at the mirror end of the tank and introducing a polarizing filter directly in the fluid, close to the stone, on the lamp side (Polaroid J-Film is only slowly attacked by many of the immersion fluids).

the polarized light system the heat must be isolated from the color filters and especially from the polarizing filter P_1, for it is extremely important that polarizing screens should not be overheated. They deteriorate (lose their polarizing property) rapidly above 60°C., and should not reach a temperature uncomfortable to the touch. Their deterioration by heat or otherwise is not usually discernible except by examining their extinction quality with the aid of a good piece of polarizing material (turned for extinction, they should pass practically no light). Since the polarizing filters and the color filters all absorb some light themselves it is important to ventilate these units, as well as to isolate the mercury lamp heat with a separate housing. The window HF_1 in the mercury lamp housing may be of one-eighth inch pyrex glass, or better the heat filter HF (specified later), and may be a moulded filter (since there is no focusing required here). However, the window for the projection lamp housing should be polished, either pyrex or heat filter as above (since here the light is used in a focused beam).

The polarizing filter P_1 and P_2 may be glass filters (polarizing film cemented between glass plates by the manufacturer) or film filters held between sheets of one-eighth inch plate-glass, with taped edges. The latter arrangement is less expensive, and the film is usually obtainable without delay. Extra filters should be kept on hand. The two filters must be mounted, relative to each other, in a crossed position (for extinction). Since light entering the tank from P_1 must be diffuse it is necessary to introduce a ground glass surface. This is accomplished without adding an extra glass plate by grinding one surface of the polarizing filter, the outer surface if a cemented glass filter, an inner surface if using loose film between glass plates (the inner surface may be used here to provide for protection from dirt). In either case the ground surface must be on the lamp side of the filter or it will depolarize the light.

In the past a carbon arc has been used as the projection light source. Such arcs are not handily turned on and off, nor adjusted, and are now difficult to obtain. An incandescent projection lamp overcomes these difficulties. A 500 to 1000-watt lamp with double-plane filament structure (filament in two planes, and staggered relative to each other, giving a solid square of illumination) is ideal for this purpose. Such lamps operate at high efficiency, are a concentrated source, have a short life, and generally require forced ventilation. A recommended lamp is the Mazda, Clear Projection, 750W—120V, T-12, C-13D Fil., Med. P.F. base. This lamp requires a small blower for ventilation and when operated with a foot switch, only during that part of the inspection that it is needed, gives a satisfactory replacement schedule.

It is important in the projection system to use large, short-focus condenser lenses, and to focus the lamp image near the center of the tank. This allows the great concentration of light which is necessary for detecting fine-textured defects, and makes it easier to determine just where in the crystal (in depth) the defect lies. Further, with the large angle of illumination available, those defects which require a specific angle of illumination may be found with less hunting. It should be noted that in figuring the object and image distances, the refractive index n of the immersion fluid must be taken into account (the window to image distance with fluid present is about n times that with fluid absent).

The color quality of the light used in the polarized light system has a considerable effect on the ease of observing the light patterns obtained when inspecting for twinning and optic axis. A typical light pattern of a piece of raw quartz viewed along the optic axis is shown in Fig. 4.8. The broad dark and light contours, "thickness-contours," are the ones used in locating the optic axis. The finely "toothed-patterns" at A, B, and C show twinning. The conditions that make the former most pronounced are not necessarily the same as those that make the latter most pronounced. The broad thickness-contours are most pronounced in monochromatic light, but barely visible in white light. This is due to the large variation of rotatory power with color, which, in all but the smallest stones, causes such overlapping of the white-light color contours as to result in practically no appearance of contours at all. This effect does not apply to the twinning regions, since in most cases the thickness of oppositely handed material is too small to develop overlapping. The result with white light is that the stone appears mainly white, except for regions of twinning where the toothed pattern is seen in color. For twinning detection, then, the advantage of white light is largely due to removal of the extraneous thickness contours. This possible advantage for the novice is not obtained without some loss of factors necessary for complete identification.

On the other hand, in determining the direction of the optic axis, the thickness contours are essential, and hence monochromatic (or a restricted spectrum) light is necessary. This illumination is most easily achieved with a mercury arc and color filters. The mercury arc emits a restricted spectrum (mainly $.578\mu$ yellow, $.546\mu$ green, $.436\mu$ far-blue lines and weak red and blue-green bands), and is very efficient. Even without filtering it gives far better thickness-contours for axis determination than does white light. The insensitivity of the eye to blue leaves mainly yellow and green. The yellow may be largely removed without appreciable loss of green by using filter F_2 and the blue and blue-green may be removed with filter F_1. However, the red can be removed only with considerable loss of green by filter F_3. The use of F_1 and F_2 alone are recommended as giving sufficient restriction of spectrum and yet high illumination. (All three filters, as used in the conoscope, give a fairly monochromatic green.) The filters need not be polished, only moulded, since here there is no focusing of light. The filters described above are the Corning glass filters:

F_1, Code 3484, H.R. Traffic Shade Yellow,

F_2, Code 5120, Didymium,

F_3, Code 4303, Dark Shade Blue Green,

HF, Code 3966, Extra Light Shade Aklo (a heat filter).

A corresponding set of filters passing more light, but giving a less monochromatic light are:

F_1, Code 3486, H.R. Yellow Shade Yellow,

F_2, Code 5920, H.R. Illusion Pink,

F_3, Code 4308, Light Shade Blue Green.

It might be added that the pronounced effects of filters are easily observed with any instrument by holding small polished filters over the eye. These same filters give some improvement even with white light, for optic axis detection. Further, if the filter were not so heavy, polished filters might be better applied at the eye than at the light source, since here they would also cut down extraneous illumination from the room. Or the same result might be obtained with large polished filters (expensive) and an eye chute at the viewing end of the system.

Several factors are of importance with regard to the immersion fluid used in the inspectoscope. The fluid should have a refractive index matching that of quartz, and be clear and colorless (to eliminate loss of light). It should be of low viscosity, so that dirt and dust may settle and air bubbles rise, rapidly (to prevent depolarization of the polarized light beam). Low viscosity also aids in the draining of oil from the stones after inspection. Water solubility of the fluid would be an aid to cleaning. Necessarily the fluid must be non-toxic and non-flammable, and preferably odorless, inexpensive and commercial. Various fluids satisfying these requirements to varying degrees have been used. Since there is no majority agreement as to which of the fluids now in commercial use is most satisfactory, no particular fluid can be recommended. (Three are listed in Chapter II, page 90.)

However, a word may be added about the required degree of refractive index match. Mineral oils of index 1.47 to 1.48 are, definitely, very poor immersion fluids for quartz. With them it is difficult to see into the interior of stones without plane polished surfaces. Ground and unpolished surfaces still cause considerable diffusion. For good inspection viewing the fluid should have an index between 1.53 and 1.56 (preferably between 1.54 and 1.55).

The refractoscope is a simple instrument especially designed for the purpose of easily and exactly checking the index match of fluid to quartz. The principle having been already noted (p. 88, Chapter II), it suffices here to describe the use of the instrument. A test tube, Fig. 4.4, filled with the fluid to be examined, is mounted in an adjustable-height stand, the optical system lowered into the test tube until the flat bottom surface of the lens contacts the fluid, and the stand is placed in an inspectoscope, conoscope, or in front of a lamp (the stand being adjusted to proper height for good illumination). The optical system comprises a lens *L* (for magnification and elimination of ripples on the liquid surface), a thin Z-cut quartz prism *Q*,[5] and a narrow slit diaphragm *S*. When the slit is viewed simultaneously through, and at the sides of the prism, a view similar to one of those shown in the figure may be seen. The two short lines are always the same distance

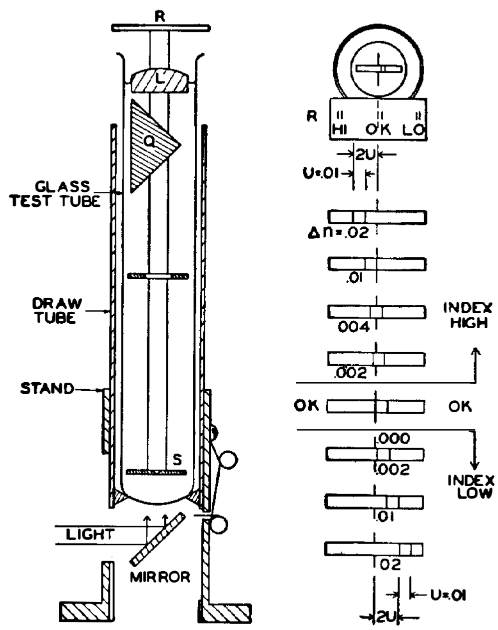

Fig. 4.4—The immersion REFRACTOSCOPE may be used in conjunction with an inspectoscope or conoscope for determining the degree of refractive index match (or mismatch) of the immersion fluid relative to quartz. At the right is shown the manner of reading the instrument.

apart and are used as a unit of index mismatch, the unit being u = .009 ≐ .01. The left-hand short line is the one to be aligned with the long line for perfect match, and the actual mismatch Δn, is measured by the separation of these two lines. The reminder tab is added to remind the operator which indications represent perfect match, too high and too low fluid index. Since the indices of both quartz and fluid vary with temperature and with color of light used, the refractoscope was especially designed for immersion directly

[5] The larger the prism angle the greater will be the sensitivity of reading, and the smaller will be the readable range of mismatch.

in the instrument using the immersion fluid. When, in such instruments, a polarizing filter is so oriented as to cut off one or both short prism-lines, these lines may be restored to view by rotating the test tube in the holder (upon continuous rotation one line and then the other will disappear). With vertical conoscopes the mirror may be removed for bottom illumination. When monochromatic light is used all three lines are the same color as the illuminant. When using non-monochromatic or white light, the long, slit line is the color of the source, while the short lines develop into two, separate spectra. In this case, that portion of the spectrum is used for alignment which is most predominantly used in the immersion instrument. The sensitivity of the refractoscope, when approaching perfect match, is about $\Delta n = .001$. This sensitivity is of course attained when one adjusts the fluid to match the quartz, by addition of the proper high or low-index component. When the fluid does not match there is a less accurate measure of mismatch, but this measure is still good for determining the degrees of fluid adjustment to be made.

The principle of the refractoscope may be even more simply applied to measuring the fluid to quartz mismatch, by making use of the inspectoscope and a basal section of quartz, using the 120° prism formed between two good, clear, adjacent natural faces. The section is placed base down in the tank at the far side from the mercury lamp, and so positioned that a vertical slit diaphragm, placed on the other side of the tank, may be viewed through the two prism faces. With the polarizing filters removed, the two images of the slit as seen through the prism do not (in general) align with the slit as seen above the prism. The image farthest from the prism vertex is the one that should be aligned with the slit, for perfect match of refractive indices. The necessity of removing the polarizing filters can be obviated by tilting the prism and slit about the line of sight, preferably at 45° from the vertical (or this might have been obviated, if the polarizing screens had been cut with their plane of polarization at 45° to the vertical).

Finally, experience indicates that the importance of keeping the immersion fluid clear and clean is not generally realized. As noted above, contamination not only gives bad scattering of the projection beam, but also depolarizes the polarized light. A perforated plate raised from the bottom of the tank is an aid in keeping the settled dirt from being recirculated again. More effective is the provision of simple, easy means for draining, filtering and refilling the tank. One or more thicknesses of chamois makes a good filter, provided the chamois is occasionally washed out with a solvent.

4.8 Photographic Study of Interior Defects

The original inspectoscope of Fig. 4.5 was used at the Hawthorne plant of Western Electric Company, in obtaining the accompanying photographs.

For inspecting twinning and direction of optic axis, the stones were viewed horizontally through the polarizing filter *E* and window, with mirror *F* removed (using mercury lamp *A*). Normally the operator looks directly down into the mirror to see the same view. For observing other interior defects, the view is from directly above the tank, through the fluid surface (with projection lamp in housing *H* being used). This is the normal manner of observation. *B* mounts heat and color filters; *C* is a polarizing filter with diffusing surface; *E*, a polarizing filter crossed to polarizer *C*; *G*, a glass window; and *IJ* is a rudimentary lamp house normally fitting over *A* to edge of *B*. The tank *D* has two rectangular windows parallel to *C* and *E*, and a circular window in rear wall for entrance of projection illumination.

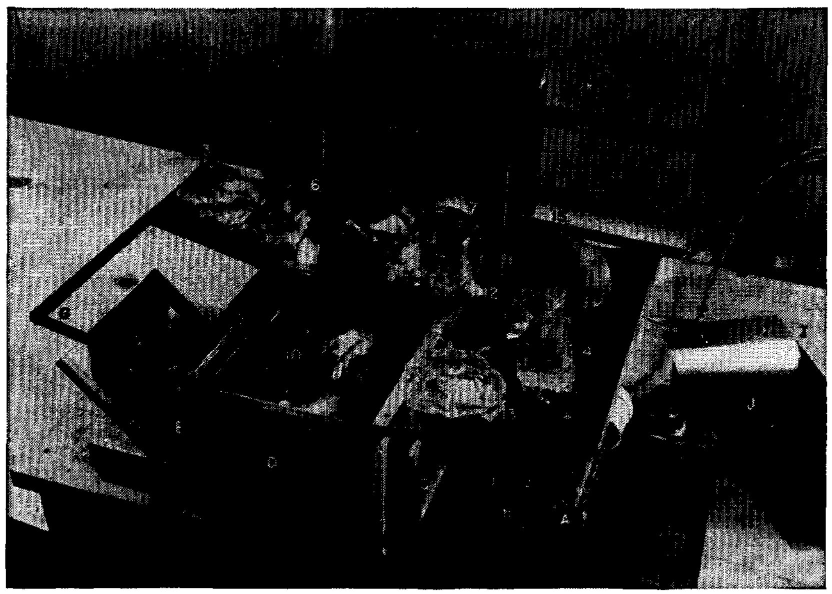

Fig. 4.5—The inspectoscope and stones used for the following figures. See also Fig. 4.3

Most of the following photographs are of stones shown, Fig. 4.5, on the drain pan and in the tank. Note that stones 3, 6, 9, 12, 14 are well faced stones; 9, 7, 13 have fractured surfaces; and 15 is typical river quartz (nearly perfect internally). The special manner of orienting the stones in the tank to obtain the desired views will appear from the following descriptions. The views are one-half to full size.

Figure 4.6 is a polarized light view of a wedge shaped basal section (one-fourth inch thick at the left, to three-fourths inch thick at the right). The wedge is viewed along the optic axis with the plane faces approximately perpendicular to the line of sight (i.e., parallel to the polarizer *C* of Fig. 4.5).

The back face is larger than the front face so that outside the borders of the front face the thickness tapers off very rapidly. Since the dark contours here show thickness of the section (as the lines of a contour map show height above sea level) one may easily determine the shape of the wedge. The inner contour *AA* is near the greatest thickness, contour *BB* intermediate thickness, and outer contour *CC* near the thinnest portions at the edges. It might be added that the thickness-contours do not exactly indicate a region of equal thickness unless the eye is distant from the stone, and unless the stone is viewed exactly along its optic axis.

Figure 4.7 is a polarized light view of an inch-thick basal section (i.e., *Z*-cut) with the parallel ground surfaces perpendicular to the line of sight

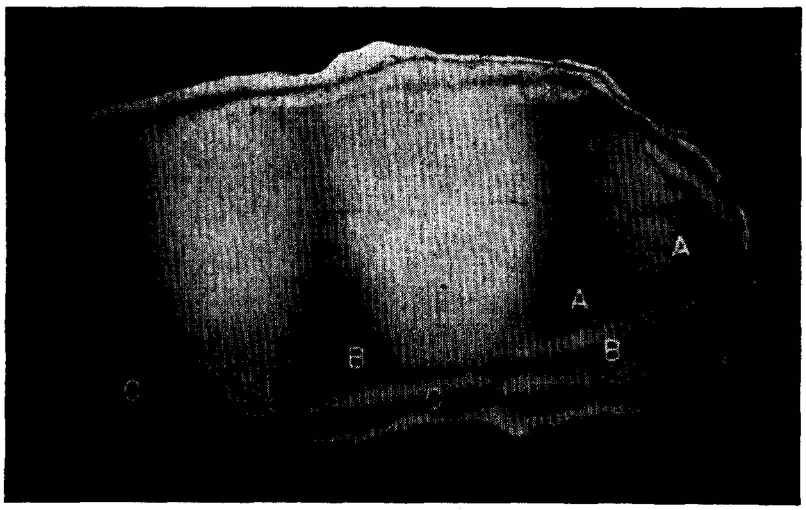

Fig. 4.6—A wedge-shaped basal section of quartz viewed along the optic axis in polarized light. The THICKNESS-CONTOURS locate regions of equal thickness, A–A thickest to C–C thinnest.

(i.e., the stone is viewed along its optic axis). Neglecting for the moment the ring pattern *C*, one observes a wide, diffuse vertical thickness-contour at *B*. With parallel surfaced stones a thickness-contour should cover the whole stone, since the thickness of the stone is uniform. Here the stone is not viewed ***exactly*** along the optic axis. If the eye be placed close these contours become circular.

Further, this view shows the effect of placing a lens between the tank window and polarizer at *E*, Fig. 4.5. The result is a ring pattern, the real image of which is at the focal distance of the lens on the eye side. The image may also be obtained on a ground glass at this point and its location is independent of the distance between quartz and lens (no rings will appear if too far separated). This ring pattern is due to conical illumination (or more correctly to conical viewing; here the illumination of the stone is diffuse). This simply illustrates the basic principle of the conoscope. The principle is further illustrated by tilting the section out of its present position, which causes the ring system to move in the direction of tilting. The theory of these effects is given in Chapter II.

Figure 4.8 is a polarized light view of a pyramidal cap of quartz with a fractured back surface (stone 3, Fig. 4.5). Here the stone is viewed along the optic axis (the six natural cap faces making equal angles with the line of sight). The continuous dark bands are thickness-contours, and again

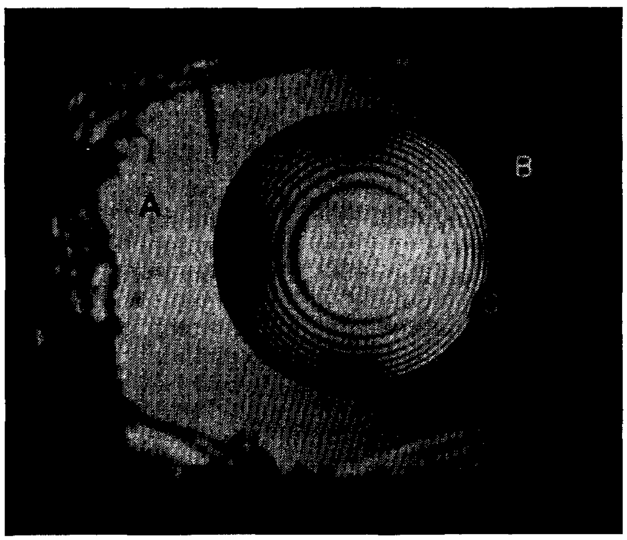

Fig. 4.7—A parallel faced, basal section of quartz viewed along the optic axis in polarized light. Note the vertical thickness-contour at B. The conoscope ring-pattern C may be obtained by introducing a lens.

represent regions of equal thickness in the optic axis direction. Had the fractured surface been flat instead of broken these contours would have been hexagonal and parallel to the hexagonal edges of the cap. The toothed-patterns at A to G are due to optical twinning (thin layers of the quartz whose handedness is opposite to that of the main stone). Although the exact shape and location are not determinable, the approximate location and extent are observed by tilting the crystal while viewing. The contour and pattern changes resulting from angularly moving the crystal (away from the position of viewing directly along the optic axis) are shown by Fig. 4.9, which should be compared with this figure.

Figure 4.9 is a polarized light view of the same stone as shown in Fig. 4.8,

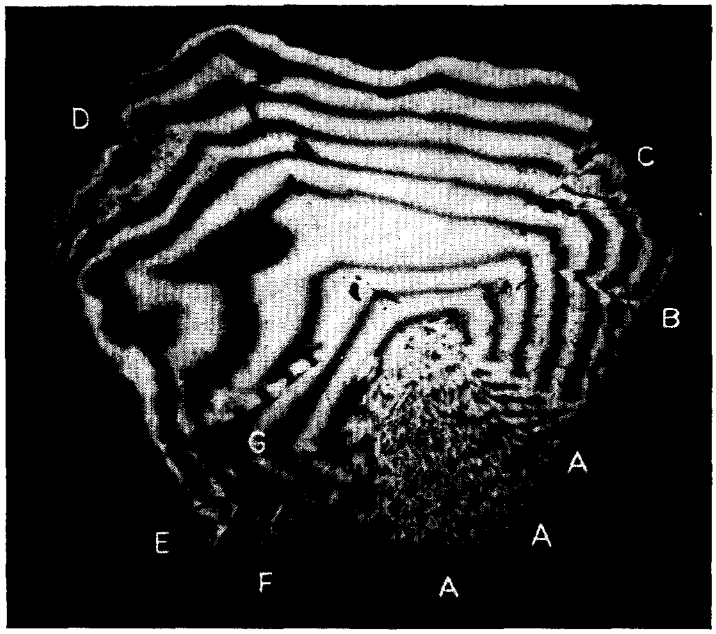

Fig. 4.8—A broken cap of quartz is viewed along the optic axis in polarized light. Few, broad thickness-contours show good alignment with the optic axis. The fine TOOTHED-PATTERNS are due to optical twinning.

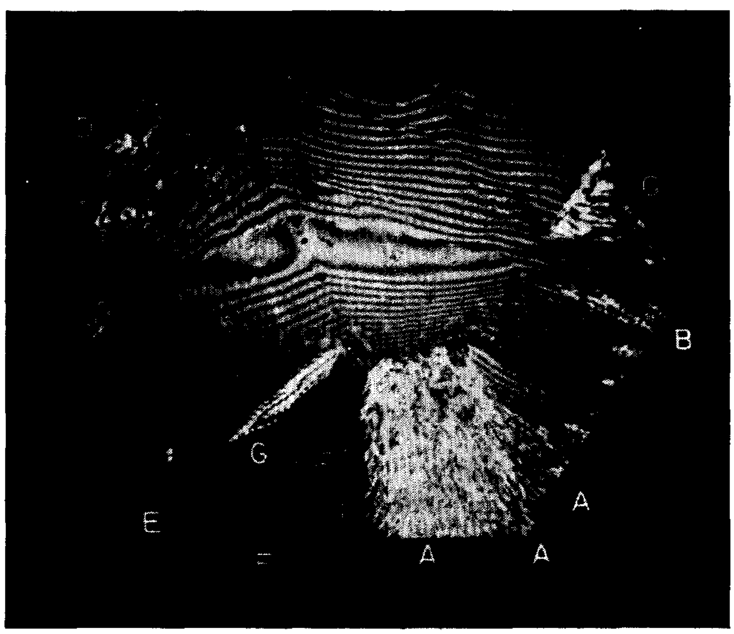

Fig. 4.9—The same cap as in Figure 4.8, but viewed slightly off the optic axis. Note the increased number of thickness-contours, and their fineness, also that some twinning regions are enhanced.

the only difference being that here the crystal is viewed slightly off the optic axis. Comparing the two views it is noted that here there are many more, and narrower, thickness-contours.[6] As the crystal is further rotated away from optic-axis-viewing, the contours multiply until they are no longer visible. Thus, it is by so positioning the stone as to obtain the fewest and widest thickness contours that the optic axis direction is determined, the axis then being parallel to the line of sight from eye to stone. It might be parenthetically noted that the handedness of raw stones may be determined in the inspectoscope, if the polarizing filter on the viewing side be rotatably mounted, by observing the contour contraction or expansion as the filter is rotated (for stones progressively thicker from outer boundary inward, the handedness rule is opposite to that for the conoscope).

A further effect of tilting the stone away from optic-axis-viewing is to enchance the toothed, twinning-patterns. Certain of these patterns are enhanced by tilting one way, others by tilting differently (note that regions *A*, *C* and *G* are much clearer here than in Fig. 4.8). Also, since the thickness-contours move about and the toothed patterns remain fixed, motion of the stone is an aid to location of twinning (except in the rare cases of large sized twins, where this does not hold). Note that optical twins usually extend inward from the original natural faces. The twin *G* which appears to be internal, actually extends inward from a cap face.

Figures 4.10 and 4.11 show projection illumination views of typical, parallel BUBBLE-PHANTOMS (in stone 7, Fig. 4.5). The light from the left converges into the stone, focuses about centrally, diverges and passes out of the stone at *B*. Due to an internal fracture in the right end the light is also reflected upward at *C*. The light beam is visible in the fluid but not in the stone, because a slightly contaminated fluid scatters far more light than does quartz.

In Fig. 4.10 the stone is held so that the phantom planes *A-A* are viewed edge on (the only way finer textured planes are visible), while in Fig. 4.11 the planes are viewed at a slight angle, to show area of the planes. Theseplanes have a texture of distinguishable bubbles. The planes are long, about an inch wide (with rectangular boundaries at their left end) and are parallel to a possible natural face (no acutal faces present on this broken stone). Such bubble phantoms are probably not permissible in any finished piezoelectric plate.

Figure 4.12 shows bubbles, cracks, veils, and phantoms (in stone 9, Fig. 4.5) and pairs of angularly joined phantom planes, *B-B*, parallel to the natural faces *A-A*; each pair forms two faces of interior phantom crystals. The texture of the phantom planes near *A-A* changes along their length from bubbly at the left to bluish at the right. A dense curved blue veil is seen at *C-C*,

[6] Actually the thickness contours are not now as closely related to the thickness as before (due to the birefringence effects being added to the rotatory effects; see Chapter II).

while a disperse, curved, bubble veil is shown at *E*. Above and to the right of *D* are two small fractures.

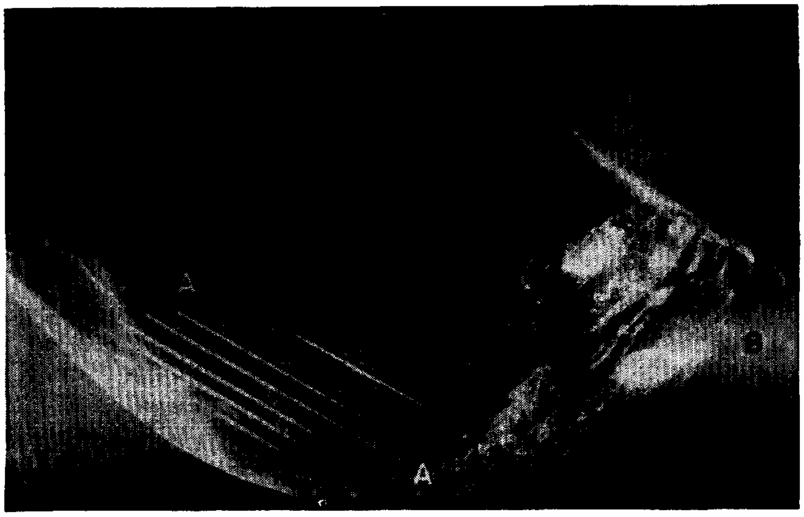

Fig. 4.10—An edge view of BUBBLE-PHANTOMS using the concentrated projection light. See also Fig. 4.11

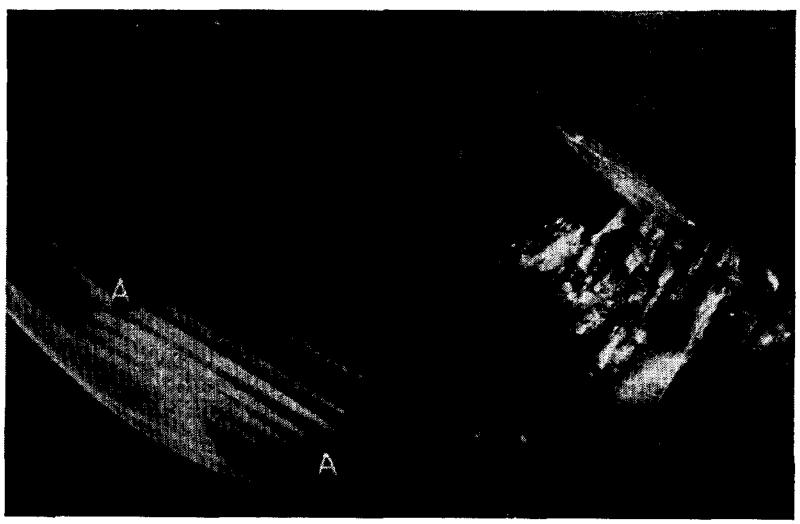

Fig. 4.11—The same stone as in Fig. 4.10, viewed at a slightly different angle. Note the bubble-texture, and the width of the planes.

Figures 4.13 and 4.14 show blue needles in two parts of a single stone which contains needles throughout (stone 10, Fig. 4.5). Only those needles which are near the concentrated focal point of the projection illumination are visible in each view. The length direction of the needles must be nearly parallel to the direction of illumination to be seen well (thus wide angle of illumination is an aid in finding the needles). Needles elsewhere in the stone may be observed by moving the stone about. The flares at the ends of the stone are due to exterior surface conditions. The needles of Fig. 4.13 are of the comet type (radiating from a point), while those of Fig. 4.14 lie in parallel groups. In each case a few of the needles are slightly feathered, and all are soft needles.

Figure 4.15 shows a stone (14, in Fig. 4.5) in which the defects are concentrated in the base, a common occurrence. Were this stone to be proc-

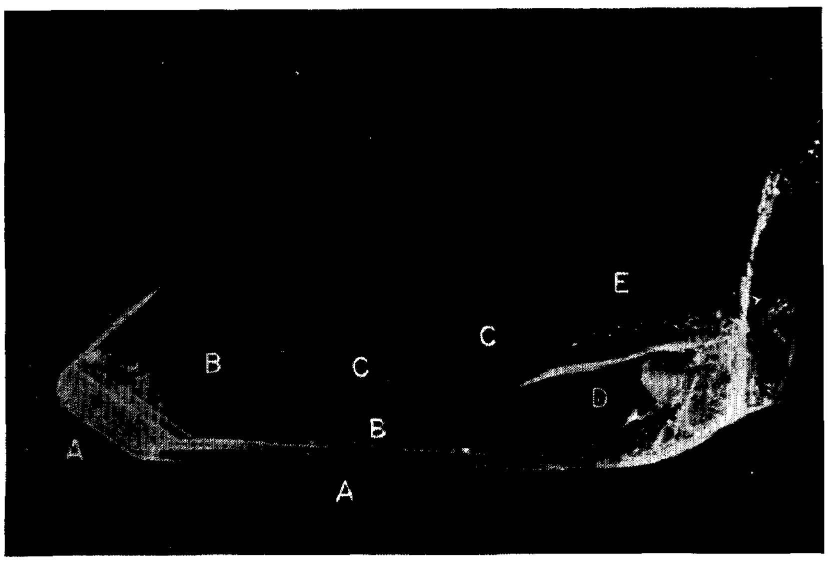

Fig. 4.12—This stone, viewed in the projection light, shows phantoms A–A to B–B, blue-veil C–C, bubble-veil E, and two fractures near D.

essed by *Z*-sectioning, a saw cut near the line *C-C* would divide the stone into a large, nearly perfect portion, and a small unusable portion which need not be further processed. Otherwise sawn, bad portions will have to be processed, or good portions of a largely bad section would be too small to obtain plates from. This points out the importance of coordinating processing with inspection, even though the stones have been already inspected and judged to be worth processing.

Bubbles and cracks fill the end of the stone at *B*, scattered bubbles appear in a veil at *A*, and a few isolated bubbles are at *D*. Note the clarity of the stone relative to the fluid, as shown by the beam of light entering the stone from the left, not visible internally.

Fig. 4.13—This stone is full of blue needles, though only those located in the focus of the light beam are visible. See also Fig. 4.14.

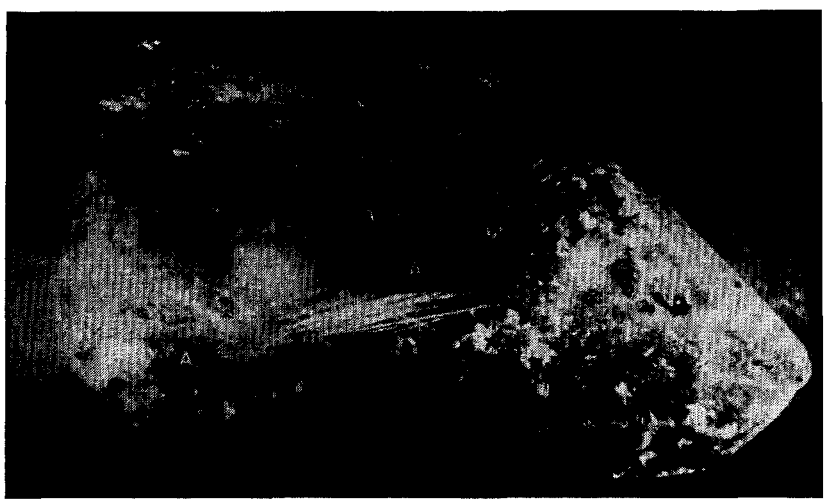

Fig. 4.14—The same stone as in Fig. 4.13 shows other blue needles in a different arrangement, by holding the stone differently with respect to the light.

4.9 Effect of Interior Imperfections on Finished Plates

The practical effect on the finished piezoelectric plates, of the various types of interior defects, is one of the least understood factors directly related to economic use of the strategic material, raw quartz. This is because of the wide variation of the types, sizes and concentrations of defects, and the further variation of the types, sizes, and requirements to be met in the finished plates. However, an analysis of the factors involved leads to some important conclusions. The various factors may be correlated as follows.

Fine textured inclusions are less objectionable than coarse textured inclusions (i.e., smokiness, and blue needles and phantoms, than white, or bubble needles and phantoms). Isolated defects are less objectionable than concentrated defects of the same texture size (i.e., isolated bubbles, than bubble phantoms). Cracks should never be permitted in finished plates. Twinning will be discussed in detail in Chapter V.

Fig. 4.15. This stone could be economically processed by cutting off the base near line C–C. A few scattered bubbles appear at D, fractures and bubbles fill the base B, and A is a bubble veil.

Further, a given defect is more likely to be tolerated in: (1) large piezoids (finished piezoelectric elements) than in small ones; (2) in low-frequency-mode plates (*CT* and *DT* types) than in high-frequency-mode plates (*AT* and *BT* types); (3) in plates to be operated at low amplitudes of vibration (filter elements, and oscillator plates with low drive) than those driven to maximum amplitudes (oscillator plates with high drive); and (4) plates having low rather than high-quality requirements (on activity, temperature-coefficient, frequency adjustment).

Thus, blue needles have long been permitted in some types of large, low-frequency-mode filter elements. While breakage has resulted from the use of blue needles in high-frequency oscillators with very high drive, it is not known that blue needles may not be used in many other types of oscillators.

It is likely that smokiness is less objectionable than blue needles (very lightly-smoky material not recognized as such has been widely used).

A further important factor, often disregarded, is related to high-frequency-mode plates and their method of manufacture, and more specifically to the method of finally adjusting the dimensions to give the required activity, frequency, and temperature characteristic. When such plates are made by the PRE-DIMENSIONING technique, which requires very small tolerances on the machined dimensions and orientation, they are finally finished by hand adjustment of only one dimension, the thickness. On the other hand, when the plates are machined to only moderate accuracy of dimension and orientation, they must be finally hand adjusted on all three dimensions to obtain satisfactory characteristics. By this method of adjustment it is possible to correct, not only for misdimensioning and misorientation, but for small defects in the quartz itself. However, with pre-dimensioning and a single dimensional adjustment practically no correction may be made for errors, or quartz defects. Thus, higher quality quartz may be necessary for manufacture by the pre-dimensioning technique than otherwise.

The conclusions that may be drawn from these considerations are: (1) only by a quantitative statistical study can it be determined whether a given type of defect will be permissible in a given type of finished plate, (2) known usability of a given type of defect in a given type of plate does not prove its usability in a different type of plate (the type includes size, mode of vibration, and required electrical operating characteristics), and (3) the method of manufacture is also related to the usability of defective quartz (i.e., pre-dimensioning vs. non-predimensioning).

Since in the past very little defective quartz has been used in the manufacture of piezoelectric elements, especially oscillators, there is little manufacturing experience that may be used as a guide to its introduction now. The quickest means of obtaining this information, and of making use of the reservoir of defective quartz, would seem to result from trial manufacture first of the most likely to succeed types of plates, from quartz with the most likely tolerable types of defects. If and when this utilization is found to be practical the less likely cases may be examined, while at the same time defective material is being used and experience is being gained in grading the raw quartz into usably defective and non-usably defective. This special grading of quartz will be difficult to control exactly. It will be easier to grade into types of imperfections than into quantities of defects per-unit-volume. Further, it will be easier in manufacturing trials to determine whether a given type of defect is permissible if the defects appear in large quantities, than if in very small quantities (where they may actually be absent in some finished plates). For these two reasons it will be preferable in trial manufacture, to select for processing stones which have the desired type of defect in large (to maximum) quantities per-unit-volume. Due to the many variables involved in both selection of material and in manufacture, a large quantity of stones must be processed for a fair trial. The criterion of usability of the defective material will then be related to number of usable finished plates (satisfying the required electrical and physical specifications on the finished plate) that can be obtained from a given quantity of raw quartz (and thus to the relative costs of producing satisfactory finished plates from defective and from non-defective quartz).

If and when a type of defect has been shown to be harmful by the above method, steps may then be taken to ascertain if some method of selection or measurement can be made on the defective raw quartz to separate the economically usable from the economically unusable material during inspection.