Crystal is subject to numerous defects, sometimes presenting a rough, solder-like, substance, or else clouded by spots upon it; while occasionally it contains some hidden humour within, or is traversed by hard and brittle inclusions. . . . Some crystal, too, has a red rust upon it, while, in other instances, it contains filaments that look like flaws. . . . *Pliny*, XXXVII, chap. 11.

THE INSPECTION AND GRADING OF QUARTZ

Samuel G. Gordon,
Associate Curator, Department of Mineralogy, The Academy of Natural Sciences of Philadelphia.

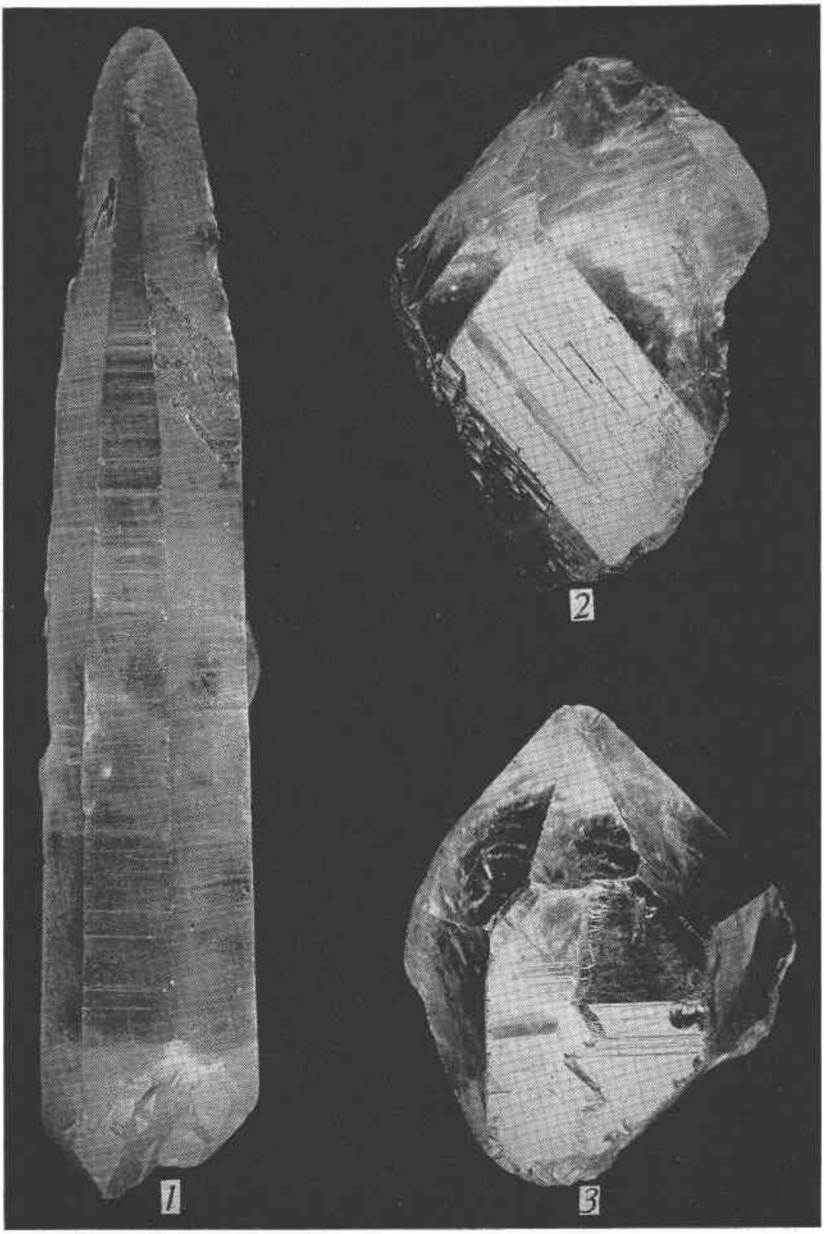
Contents

Abstract

To be usable for radio frequency oscillators, a quartz crystal must have part of its volume free of twinning and detrimental inclusions. Dauphiné ("electrical") twinning may sometimes be detected upon the surface of the crystal, or by etching crystals or sections. Brazil ("optical") twinning, intergrowths of right and left quartz, is easily detected by viewing a crystal, immersed in oil, and between crossed polaroids, in the direction of the optic axis. An oil bath, so set up, has been called a quartz inspectoscope. It also permits the quartz to be scanned in an intense beam of light for cracks, bubbles, inclusions, and the inexplicable oriented linear clouds in V-shaped clusters called "blue needles." The apparatus is not only useful in grading quartz for "usability," but for inspection of sections, bars, wafers, and blanks to avoid useless processing. A portable field model, using sunlight, is described. An example of the rare type of twinning according to the "combined laws" is illustrated.

Introduction

The usefulness of quartz for radio-frequency oscillators depends upon the absence of deleterious matter and structures that affect its elasticity or cause electrical anomalies.



FIGS. 1–3. Typical Brazilian crystals of radio grade. Fig. 1: a "candle"; Fig. 2: a crystal with a large *s* $(11\bar{2}1)$ plane, showing characteristic striations parallel to edge *s*:*r*; Fig. 3: a Dauphiné ("electrical") twin, showing the characteristic sutured boundary on *m* $(10\bar{1}0)$.

We are not particularly concerned with the obvious inclusions of rutile, tourmaline, hematite, pyrite, mica, chlorite, or moving bubbles that are of interest to mineralogists but with quite common defects that occur in *eye-clear* rock crystal. Twinning is as prevalent in quartz as it is in plagioclase feldspar, and quartz suitable for cutting flawless crystal balls may consist of polysynthetic twinned laminae that destroy its usefulness for radio purposes. Limpid quartz is apt to be filled with invisible bubbles, or the remarkable linear clouds in V-shaped clusters called "blue needles."

Quartz oscillators may be cut from either left or right quartz, but the presence of both in the same plate results in electrical anomalies. Since quartz oscillators are cut in a definite crystallographic orientation, a plate oriented with respect to one part of a twin will be misoriented for the other.

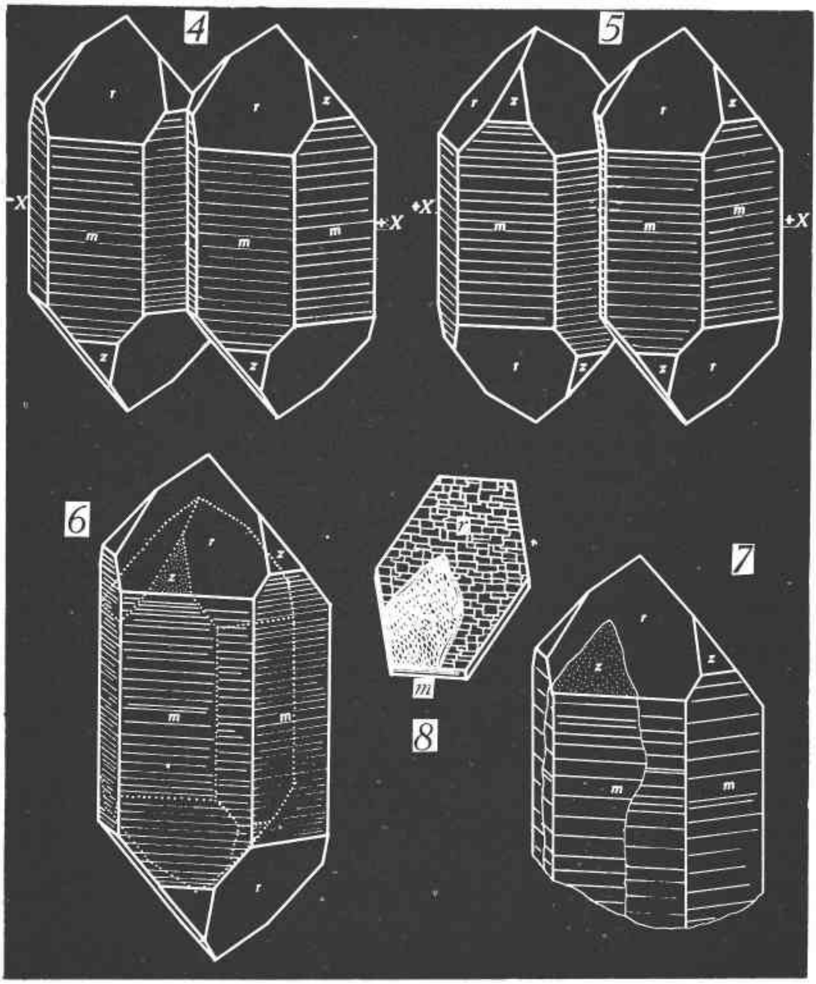
The presence of some minute bubbles and "blue needles" may be tolerated in large plates vibrating at low frequency (such as X-cuts and filter plates), but it is obvious that a bubble in a small plate will occupy an appreciable volume of the whole plate and may seriously affect its elastic properties. It is now the general practice to etch oscillator plates to their nominal frequency-thickness; bubbles and "blue needles" are rapidly attacked by the etching solutions, and their absence should be assured.

It is not only necessary to grade the quartz on purchase, but to inspect it at various stages of manufacture to avoid useless processing. It is also possible to plan the cutting operations to get a maximum yield from twinned quartz, and rules to accomplish this will be given in an accompanying paper.

Twinning in Quartz

Untwinned quartz is quite rare, and it is unusual to find a crystal which is not twinned according to both the Dauphiné and Brazil laws.

Dauphiné ("electrical") twinning is the designation of Dauphiné twins by radio engineers: an intergrowth of either two right *or* two left individuals, at 180° to each other, with *c* as the twin axis (Figs. 4–8). The rhombohedral faces *r* $(10\bar{1}1)$ and *z* $(01\bar{1}1)$ become coplanar, as do also *m* $(10\bar{1}0)$ and $\bar{m}$ $(\bar{1}010)$. Such twins are often recognized by the sutured boundary on *m* $(10\bar{1}0)$, or by a difference in the sheen of the coplanar forms *r* and *z* on the apex faces (Fig. 9). Dauphiné twins are also recognizable by the presence of *s* $(11\bar{2}1)$, and *x* $(51\bar{6}1)$ on both edges of a prism face, when the normal, untwinned trigonal symmetry calls for these faces only on alternating prism edges. This type of twinning is



FIGS. 4–8. Parallel growth and Dauphiné ("electrical") twinning. Fig. 4: a parallel growth; Fig. 5: a Dauphiné twin: an intergrowth of two right *or* two left crystals with the polarity of their X-axes reversed; Fig. 6: an idealized intergrowth of the individuals of a Dauphiné twin showing *r* and *z* as coplanar; Fig. 7: a more natural representation; Fig. 8: appearance of an etched wafer showing the characteristic "ripple" and "shingle" structure developed by coplanar *z* and *r*, respectively.

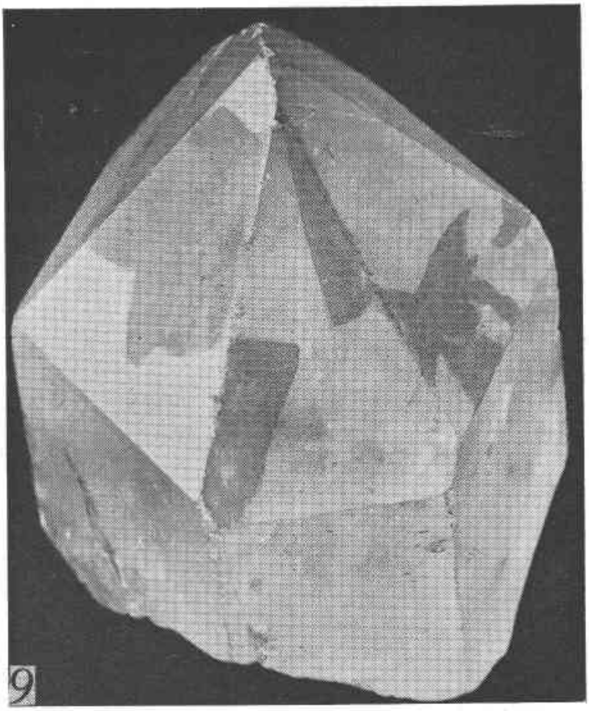
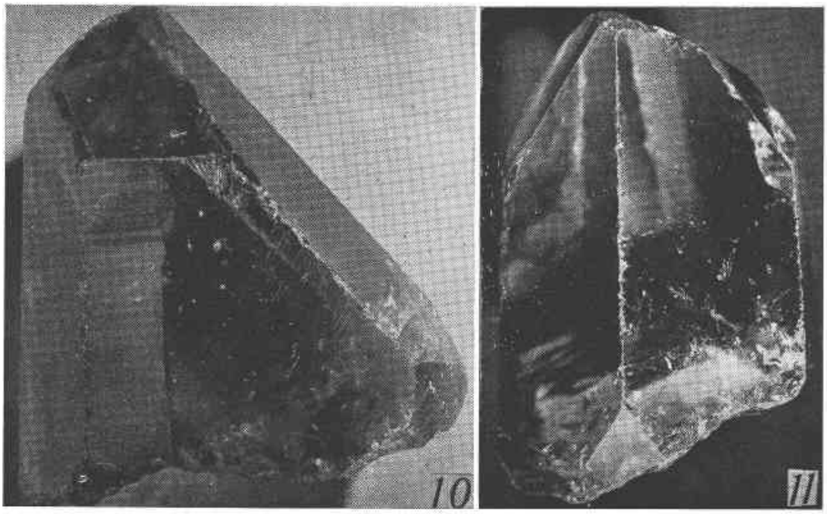


FIG. 9. In Dauphiné ("electrical") twinning, *r* (10$\bar{1}$1) and *z* (01$\bar{1}$1) are coplanar, but often distinguishable by differences in sheen, due to natural etching. (Photograph through the courtesy of Lieut. William B. Gray, Jr.)

called electrical twinning because it results in a reversal of the polarity of the electrical axis (X [11$\bar{2}$0]) at the twin boundary.

In marked contrast to twins of the Brazil law ("optical twinning") the twin boundaries of Dauphiné twins are quite irregular, and this serves to distinguish the two in etched sections (Figs. 12–13).[1] In Dauphiné, "electrical" twins, the coplanar areas, *r* and *z* can be distinguished in etched wafers by their difference in translucency and structure; *r* developes a characteristic "shingle" structure, while *z* develops a less symmetrical "ripple" pattern (Fig. 8); this is further discussed in the following paper.

[1] Leydolt, Franz, Über eine neue Methode, die Struktur und Zusammensetzung der Krystalle zu untersuchen, mit besonderer Berücksichtigung der Varietäten des rhomboedrischen Quarzes. *Sitzber. d. kais. Akad. d. Wiss. Wien* (Math.-Natur. Classe) XV (1) 59–81, **1855.**



FIGS. 10–11. Two views of a Dauphiné ("electrical") twinned crystal, showing a sutured boundary on the prism *m* $(10\bar{1}0)$, and the equal development of two apex faces and zonal development of these with *m* $(10\bar{1}0)$. The latter, resulting in a pseudo-prismatic appearance of the apex faces with long edges in contact with *m* is diagnostic in detecting electrical twinning. (Photographs by NAP.)

FIGS. 12–13. Etched sections reveal twinning strikingly, and Dauphiné ("electrical") twinning can be distinguished from Brazil ("optical") twinning by the irregular boundaries developed in the former (Fig. 12) in contrast with the straight boundaries developed in optical twinning (Fig. 13). These sections are perpendicular to the optic axis. (After Leydolt.[1])

A Dauphiné twin may consist of a pair with an irregular boundary between them. Other crystals contain many quite irregular patches—which suggests that the twinning may be secondary; that is, due to stresses set up through sudden changes of temperature and pressure. A small plate heated in an oven (not necessarily to the inversion point) will twin on sudden cooling, and etching will reveal curved boundaries symmetrical to the edges of the plate.

Brazil "optical twinning" is the name applied to twinning after the Brazil law, because it is readily detected by examination in polarized light. This kind of twinning is an intergrowth of the two enantiomorphous forms: of right and left individuals.[2]

The story of the discovery of this kind of twinning is recorded by Sosman:[3]

"Twinning in quartz was first placed on record by Weiss[4] in 1816. He described and figured clearly the orientational (Dauphiné) type of quartz twins. The chiral (Brazil) type he appears also to have observed, though rarely, but spoke of it cautiously as a kind of intergrowth which might possibly be explained as a case of twinning allied to the orientational. The difficulty was simply that the difference between the two alternative choices,—positive versus negative forms on the one hand, and right versus left on the other,—had not been made clear.

"Herschel[5] happened upon some twinned crystals in the course of his pioneer work on the correlation of optical activity and face development in quartz and was puzzled to find one specimen which had trapezohedral faces perfectly distinct and in contact but 'tending opposite ways around the summit.' Unfortunately it was 'in the possession of Mr. Brooke,' and Mr. Brooke apparently valued it too highly to let it be damaged in the interest of science, so Herschel was not allowed to section it and test its rotatory power for polarized light. The sympathy of every true mineral collector will be with Mr. Brooke, even though it did postpone an important scientific discovery for twenty-five years.

"The explanation for this peculiar crystal was not forthcoming until G. Rose[6] published his comprehensive work in 1846. He was the first to show that the apparently homogeneous crystal with the opposite tending faces is really a compound crystal, twinned according to the Brazil law."

[2] Lord Kelvin (Baltimore lectures, 1904, p. 640) called it *chiral* twinning (from the Greek, for hand). The optical rotary power due to handedness has been called *chirality*.

[3] Sosman, Robert B., *The Properties of Silica*, p. 196–197, Chemical Catalog Co., New York (1927).

[4] Weiss, C. S., Über den eigenthümlichen Gang des Krystallisations-systemes beim Quarz, und über eine an ihm neu beobachtete Zwillingskrystallisation: *Ges. Nat. Freunde Mag. Berlin* **7**, 163–181, pl. 4 (1816).

[5] Herschel, J. F. W., On the rotation impressed by plates of rock crystal on the plane of polarization of the rays of light, as connected with certain peculiarities in its crystallization: *Trans. Cambridge Phil. Soc.* **1**, 43–52 (1821).

[6] Rose, Gustav, Über das Krystallisationssystem des Quarzes: *Physik. Abh. Köngl. Akad. Wiss. Berlin*, 1844: 217–274, pl. 1–6 (1846).

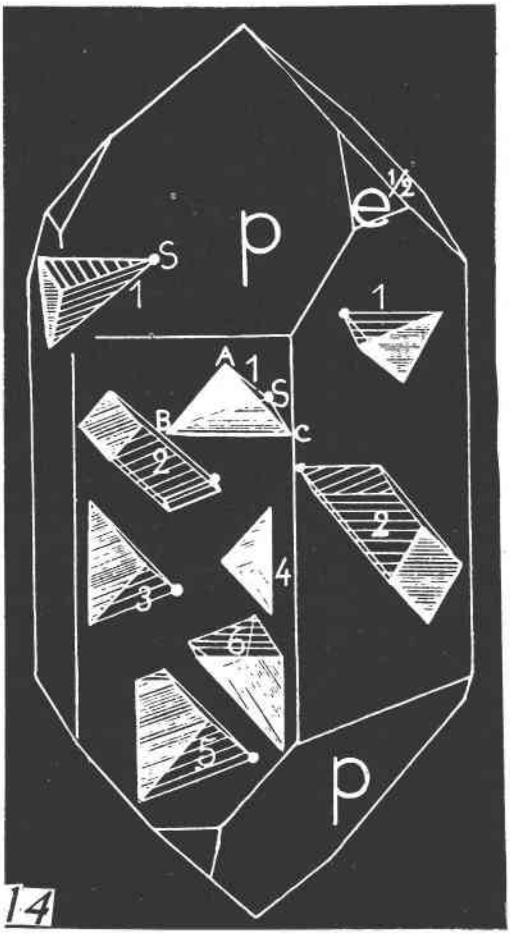


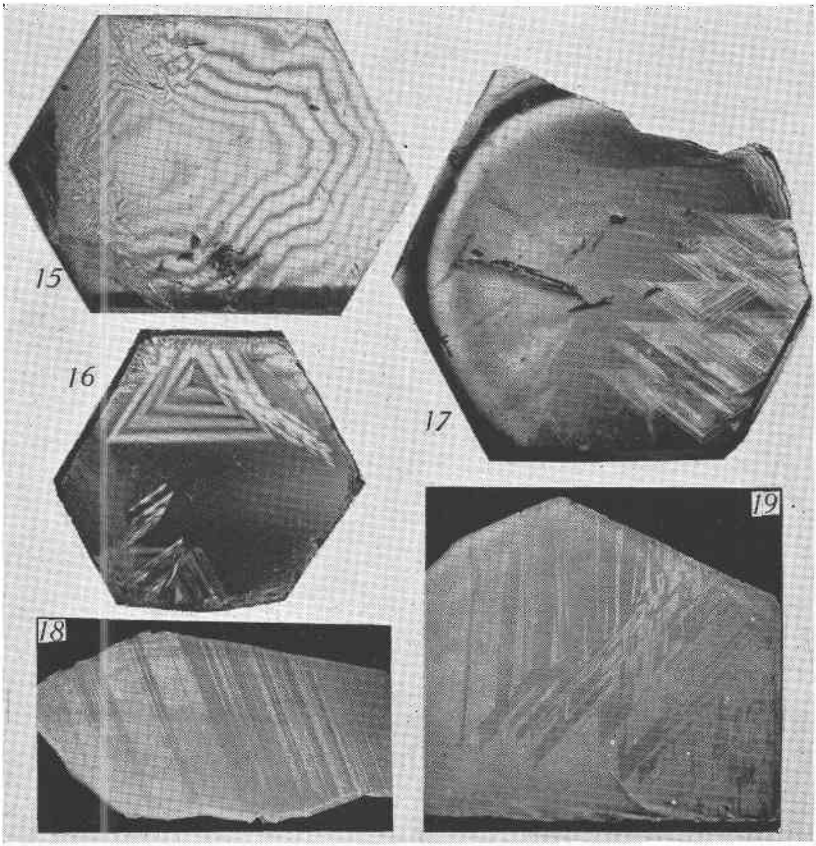
FIG. 14. Brazil ("optical") twinning is the intergrowth of right and left quartz. It consists of laminae of one hand enclosed within a host crystal of the opposite hand. The remarkable shapes assumed by these "satellites" are shown in the idealized perspective of the Abbe C. Gaudefroy,[7] who listed as the principal forms $m(e^2)$ $10\bar{1}0$, $r(p)$ $10\bar{1}1$, $z(e^{1/2})$ $01\bar{1}1$, s $11\bar{2}1$, and $c(a^1)$ 0001.

[7] Gaudefroy, C., Sur les groupements de cristaux de quartz à axes parallèles: *Bull. soc. franç. min.* **56,** 5–63 (1933).

Lamellar twinning was discussed by John W. Judd: Additional note on the lamellar structure of quartz crystals, and the methods by which it is developed: *Min. Mag.* **10,** 123–135 (1892).

Similar twinning in cinnabar was described by Waldemar Lindgren: *U. S. Geol. Surv. Bull.* **61** (1890).

Crystals twinned according to the Brazil law and resembling the idealized drawing of Rose, which has been copied in all text-books, are rare, and their existence requires confirmation. This type of twinning is seen rather as a growth of laminae of one hand within a host crystal of the



FIGS. 15–19. Brazil ("optical") twinning may be detected by viewing crystals (immersed in oil between crossed polaroids) in the direction of the optic axis. The contour of the base or termination is seen in bands of color expressing various degrees of rotation of the plane of polarization because of varying thickness of quartz (Fig. 15). Laminae of the opposite hand are seen as triangular areas (Figs. 16–17). Etching of sections localizes the twinning (Figs. 18–19). (Photographs 15, 18–19 by NAP.)

opposite hand (Figs. 14–19). The shapes assumed by these inclusions are remarkable: they suggest crystals of the pedial class of the triclinic system, in that they are triangular polygons, and often with but one form of a kind. Not only are they of a habit entirely different from that assumed by the host crystal, but they may show a well developed basal plane! The form of these "satellites" was described by the Abbé C. Gaudefroy[7] in 1933, then president of the Sociéte Française de Mineralogie, and the principal types are reproduced in his idealized perspective (Fig. 14). As a rule the laminae are quite thin, and many laminations of apparently the same dimensions may occur rhythmically spaced above one another in the host, and invariably parallel to the major rhombohedral faces (*r* $10\bar{1}1$) (Fig. 17).

Most quartz crystals are intimate intergrowths of polysynthetic laminae of right and left quartz, and it is principally in Brazil that quartz crystals occur relatively free of the twinning to which that country has given its name! Quartz of radio quality from Brazil usually shows the parasitic laminae at the margins of the crystals, and the interior may be quite free of them.

Twinning According to the Combined Laws

Electrical twins (Dauphiné law) are described as two right, *or* two left individuals, at 180° to each other with respect to the theoretical twin plane $11\bar{2}0$; the faces *r* ($10\bar{1}1$) and *z* ($01\bar{1}1$) are coplanar. Optical twins (Brazil law) are simply intergrowths of the two left and right enantiomorphous form: the forms *r*, *z*, *m* on one individual are parallel to *r*, *z*, and *m* of the other.

An example of the combined laws[8] is shown in Fig. 20. Two parallel planes perpendicular to the electrical axis (*a*, X-axis) have been cut on the crystal; one was etched and the other polished: the photograph shows the view looking through the polished (+) X-plane to the etched (−) X-plane at the back.

From the parallelogram light figures (described in the accompanying paper on Orientation), the two parts may be considered as an electrical twin of a right and a left crystal, or as an optical twin in which the right and left crystals are 180° to each other, with *r* and *z* coplanar.

It is characteristic of electrical twins to have irregular boundaries, while optical twins have straight boundaries: it will be noted that this specimen shows both; the boundaries parallel to *r* ($10\bar{1}1$) are plane, as it should be in an optical twin: the other boundary is quite irregular.

[8] Found by Mr. Isaac Herr of the Commercial Crystal Company of Lancaster, Pa. Other examples, discovered by Mr. Hoyt Brubaker, were generously given to the writer by Mr. P. R. Hoffman of Carlisle, Pa., for study. L. A. Thomas, *Nature*, **155**, 425 (April 7, 1945) suggests the name "compound optical twinning" for the combined laws.

Detection of "Optical" Twinning in Polarized Light

Twinning according to the Brazil law is called "optical" twinning because it is so readily detected by viewing the quartz between crossed polaroids in the direction of the optic axis. This may be readily demonstrated by immersing a *clear* quartz crystal in a beaker or culture dish filled with an oil of proper index. The beaker is set upon a pair of glass

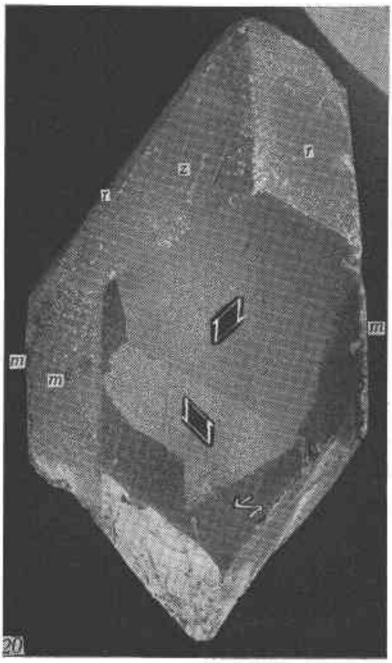
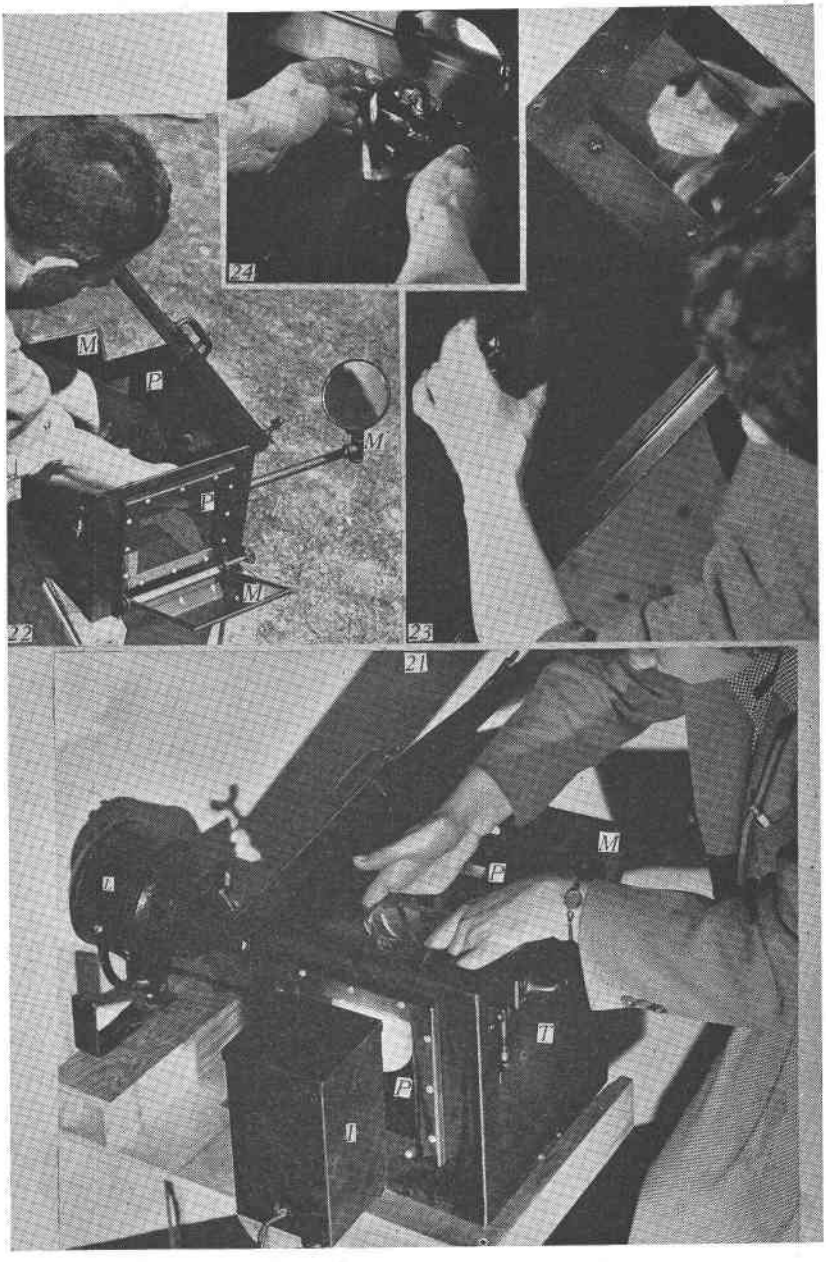


Fig. 20. A crystal twinned on the rare "Combined Laws." View perpendicular to electrical axis [$11\bar{2}0$]: through the polished face [$+X$] to etched face in back [$-X$]. Sketched in are the light figures seen on the etched surface on viewing a point source of light. The central area has the character of both an optical twin (straight boundaries parallel to *r* ($10\bar{1}1$)), and of an electrical twin (the irregular boundary). About 2/5 natural size.

plates between which is a film of polaroid (a diffusing ground glass plate is also desirable). The plates are placed upon a laboratory tripod—an electric light within the tripod may be used for illumination. A second polaroid, crossed to the first, is set upon the beaker. Upon viewing the quartz crystal at a slight angle to the optic axis, the twinned laminae will be seen as thin, spectacularly colored plates (Figs. 15–17). The phe-



FIGS. 21–24. The quartz inspectoscope is simply an oil bath in which a quartz crystal can be viewed between crossed polaroids to detect optical twinning; it also permits scanning of the crystal in an intense beam of light for flaws and inclusions. Fig. 21: the Klieg model used in the plant: the tank (T) has a pair of crossed polaroids (P), a light source (I) and a view-

nomena is striking only if viewed within a few degrees of the optic axis, and it is essential that the quartz be immersed in an oil approximating the indices of refraction of quartz .The most satisfactory liquid is ortho-free tricresyl phosphate ($n=1.55$) sold under the trade name of *Lindol.*[9]

In general, the laminae have the appearance of right triangles: with the base of the triangle parallel to the prism face with which it is usually in contact; the hypotenuse parallel to a prism face at 120° to the first (parallel to an $m{:}r$ edge, or an X axis); and the third side parallel to an $r{:}r$ edge (Y axis; prism normal). Other plates appear as equilateral triangles, with all three sides parallel to traces of prism faces ($m{:}r$ edges).

The usefulness of this method depends upon the fact that the parasitic laminae are thin and wedge-like, and they rotate the plane of polarization in the opposite direction to that of the host. The polysynthetic twin laminae are thin enough to show several orders of interference colors, while the host is so thick that interference colors are seen only as colored bands fringing the base of the crystal and expressing variations in thickness of the contour of the latter (Fig. 15). The interference colors shown by various thicknesses of quartz are tabulated in Table 1.[10]

The Quartz "Inspectoscope"

The quartz inspection apparatus used in crystal-cutting plants is essentially a tank in which the quartz can be immersed in oil and examined in polarized light, as well as in the intense beam of a spot-light (Figs. 21–24). Often it was merely a fish aquarium, the edges of which had been

[9] Obtainable from the Celanese-Celluloid Corporation, 180 Madison Avenue, New York City, for about $0.285 per pound in 5 gallon lots. Specify *ortho-free*, a grade which seems to be free of dermatological hazards. After immersion, crystals are readily cleaned with laundry soap and water.

[10] More than a century ago, Fresnel showed that quartz was birefringent in the direction of the optic axis. Sosman quotes for ω and ϵ, respectively 1.5441887 and 1.5442605, or a birefringence of 0.0000718. Plane polarized light traveling in the direction of the optic axis is separated into two circularly polarized rays, of opposite sign and different speeds, which emerge to form plane polarized rays with the interference color composed of the resultants of the rays which were not extinguished. Airy showed that rays not parallel to the optic axis were transmitted as two sets of elliptically polarized waves. G. Szivessy (*Fort. Min. Krist. Petr.* **21,** 111–168 (1937)) has shown that α quartz is optically inactive at 56°10′ to the optic axis. Rays traveling perpendicular to the optic axis are plane polarized. Full discussions of the rotatory power of quartz will be found in the works of Sosman and Tutton.

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ing mirror (M); (L) is an intense spot-light. In the field, a circular mirror is used to reflect the direct rays of the sun for scanning the crystal for flaws and inclusions (Fig. 22), while another mirror (M) is used to reflect light from the sky overhead in searching for optical twinning. Fig. 23: the crystal is being viewed in the mirror (along the optic axis direction) for twinning. Fig. 24: scanning the crystal in an intense beam of light for inclusions. (Photographs, courtesy of NAP.)

sealed with asphalt paint, and which had been fitted with screens of polaroid at the ends, similarly sealed against seepage of oil. Illumination was provided by a pair of 40 watt electric light bulbs, diffused by ground glass or opal glass at one end, and a mirror was provided for viewing the quartz at the other end.

TABLE 1. ROTATION OF THE PLANE OF POLARIZATION OF QUARTZ*

Color extin-guished	Fraun-hofer line	Wave length λ in μμ	Rotation per mm. at 20°C.	Thickness necessary to rotate 90°	Thickness necessary to rotate 180°	Interference color between crossed nicols
Red	A	760.4	12.65°	7.15	14.30	Green
	a	718.36	14.30	6.29	12.58	Blue
	B	686.71	15.75	5.71	11.42	
Orange	C	656.21	17.31	5.19	10.39	
Yellow	D_2	589.513	21.69	4.14	8.29	Violet
	D_1	588.912	21.725	4.11	8.23	
Green	E	526.913	27.54	3.26	6.53	Red
Blue	F	486.074	32.76	2.75	5.49	Orange
Indigo	G	430.725	42.59	2.10	4.20	Brownish yellow
Violet	h	410.12	47.49	1.89	3.79	
	H	396.81	51.19	1.75	3.51	Yellow

* Johannsen, A., *Manual of Petrographic Methods*, p. 109. McGraw-Hill, New York (1914).

Such equipment was modeled after the quartz "inspectoscope" developed by the Bell Laboratories for the Western Electric Company, where mercury vapor lamps were used for illumination, and the light was filtered through a series of Corning color filters including an Illusion pink, a yellow shade yellow, and a Light Shade blue green, as well as a heat filter. This instrument had advantages over direct viewing tanks used in the Bureau of Standards, where the lower polaroid plate was in the vulnerable position of being at the bottom of the tank and was soon obscured by dirt or cracked by quartz crystals slipping from the operator's hand: moreover, the mirror image seemed to present a better picture of the twinning that could be gathered from scanning the crystal directly.

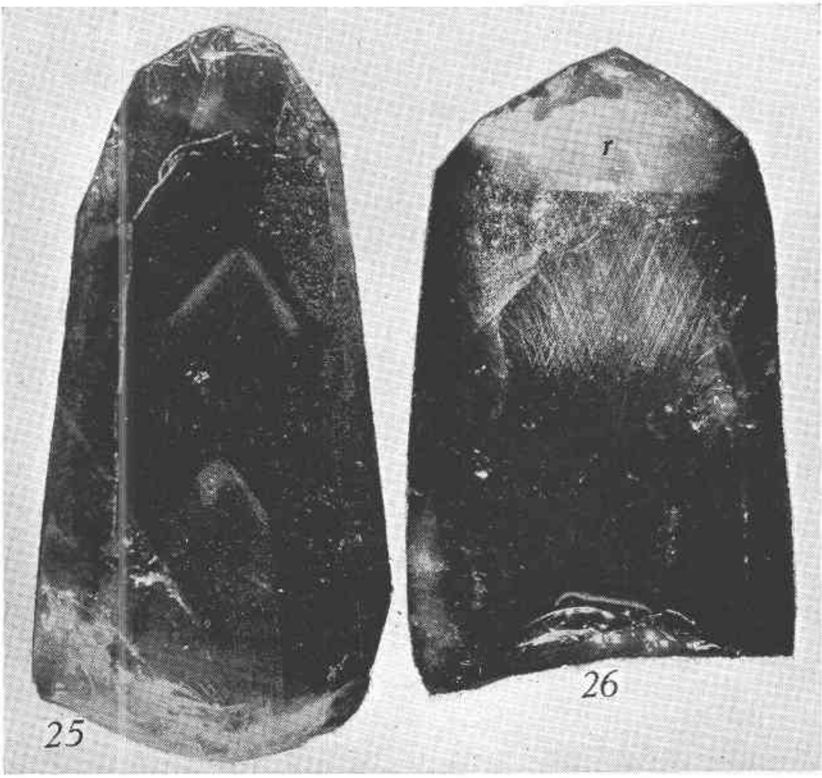
Field Model

A smaller, portable apparatus (Fig. 22)[11] was introduced for use in the field, but proved equally popular in crystal-cutting plants. It was designed for use in regions remote from electricity, and was illuminated by

[11] Manufactured by Kliegl Brothers, 321 West 50th Street, New York City: a Klieg spotlight is provided for plant use.

light reflected from the sky and the sun by a pair of folding mirrors: for that reason they became known in Brazil as "sun buckets." A tight fitting lid permitted transportation of the oil in the bucket.

Upon reaching a prospect, the tank is set upon a rock, with the sun to one side of the prospector (Fig. 22). The lid is unclasped and set up as a shield to exclude extraneous light. The mirrors are pulled out and turned



Figs. 25–26. Eye-clear, limpid quartz is quite apt to show phantoms or "blue needles" on scanning the crystal, immersed in oil, in an intense beam of light.

to their reflecting angles. The light of the sky overhead or in front of the prospector is reflected into a polaroid window, and the mirror opposite it is adjusted for viewing quartz for optical twinning. Another, circular, mirror is set to reflect the sun's rays into the tank for examination for flaws, bubbles, and other defects: a metal shield hung over the tank can be slid over this window to exclude the direct beam during the examination in polarized light.

Detecting Optical Twinning in Practice

The quartz must be thoroughly cleaned before inspection; moreover cloudy areas at the sides or base must be trimmed by means of a hammer, rock trimmer or saw: this is particularly true of such crystals as occur at Hot Springs, Arkansas in which the lower part is apt to be quite cloudy.

The clear crystal is held in the oil bath, with the optic (Z) axis direction (*c*), perpendicular to the crossed polaroids. The irregular base will be fringed by concentric color bands expressing variations in thickness of the margin of the crystal (Figs. 15–17). Twin laminae will appear as colored *triangular* blades along the side or shooting through the crystal, or cutting across the concentric color bands of the margin of the mass. By slightly tilting the crystal the position of the twinned areas can be approximately located and usable volume of the quartz, free of twinning, can be estimated.

Since it is necessary to orient the quartz precisely in order to see the twinning, some difficulty may be experienced at first with defaced quartz; the following rule will be helpful: Turn the mass horizontally in the oil bath until the color bands concentric with the margin appear or the quartz is at extinction; in the latter case, rotate it now about an axis parallel to one of the polaroid vibration directions.

Flaws and Inclusions in Quartz

An intense beam of light from a carbon-arc or a projector is used to detect flaws and inclusions in quartz. The quartz is scanned while it is submerged in an oil approximating it in index of refraction, and the effect is as revealing as the beam from a moving-picture projector through a dust-laden darkened room.

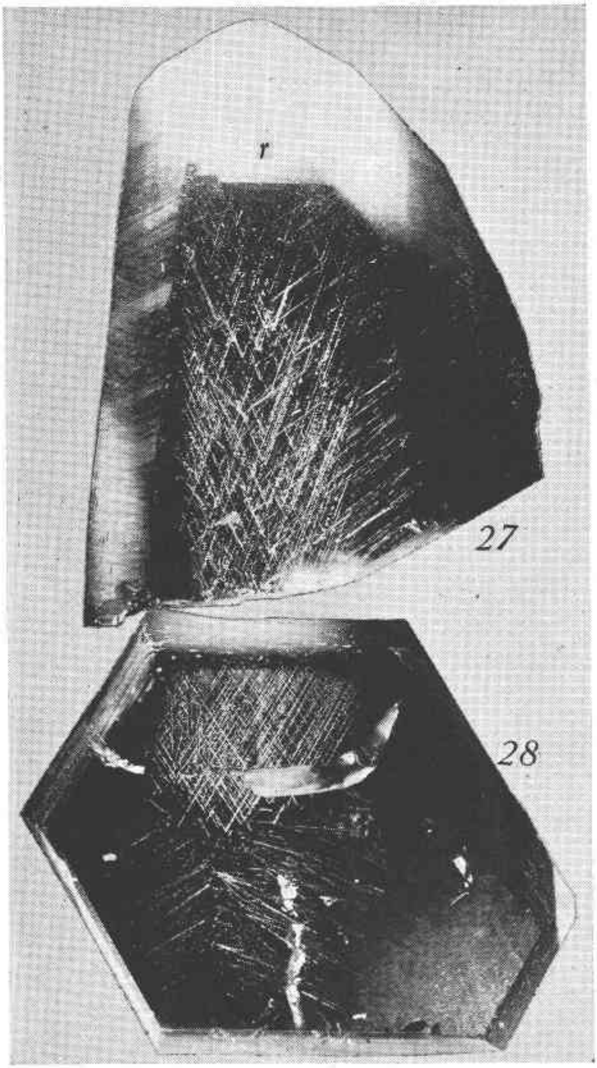
The principal defects are fractures, cavities, bubbles, "ghosts," and the remarkable "blue needles" and "blue feathers" (Figs. 26–28). Various designations used by quartz inspectors are:

(1) "Bubbles": bubble-like cavities, distinguished as "large" or "fine."

(2) "Veils," "heavy" or "fine": more or less continuous sheets of small bubble-like cavities.

(3) "Clouds" and "haze": aggregates of fine, bubble-like cavities.

(4) "Ghosts" are the familiar "phantoms" of mineral collectors (Fig. 25): earlier growths within the crystal, which become visible when a beam of light is reflected from the minute fractures or parting planes which outline them. They are also seen as alternating colorless and smoky zones within the crystal.



FIGS. 27–28. "Blue needles" or "blue feathers" are quite common inclusions of linear clouds in V-shaped clusters, with a preferred orientation of the limbs of the V parallel to an $r:z$ intersection edge, and the plane of the V parallel to an r face. The apex of the V always points to the base of the crystal, never to the terminated end. The reticulation is only apparent, the V's being at successive, often equidistant, levels. They seem to be systems of minute parting planes. (About 3/4 natural size.) The relation of the basal view (Fig. 28) to Fig. 27 is as follows: the edge which is common to the two is in contact, and the exact relation can be seen by folding Fig. 28 under Fig. 27 along this edge. (The prism face and basal section shown were kindly polished by William Van Horn.)

(5) "Blue needles" and "blue feathers" (Figs. 26–28): these are inexplicable linear clouds which appear bluish because of selective absorption of rays of others colors. They occur as pairs of needles forming a V, with the apex of the V *always* towards the base of the crystal, and the two arms of the V have a preferred orientation parallel to *r*:*z* edges; in other words, a V outlines a plane parallel to *z* towards the base of the crystal.[12]

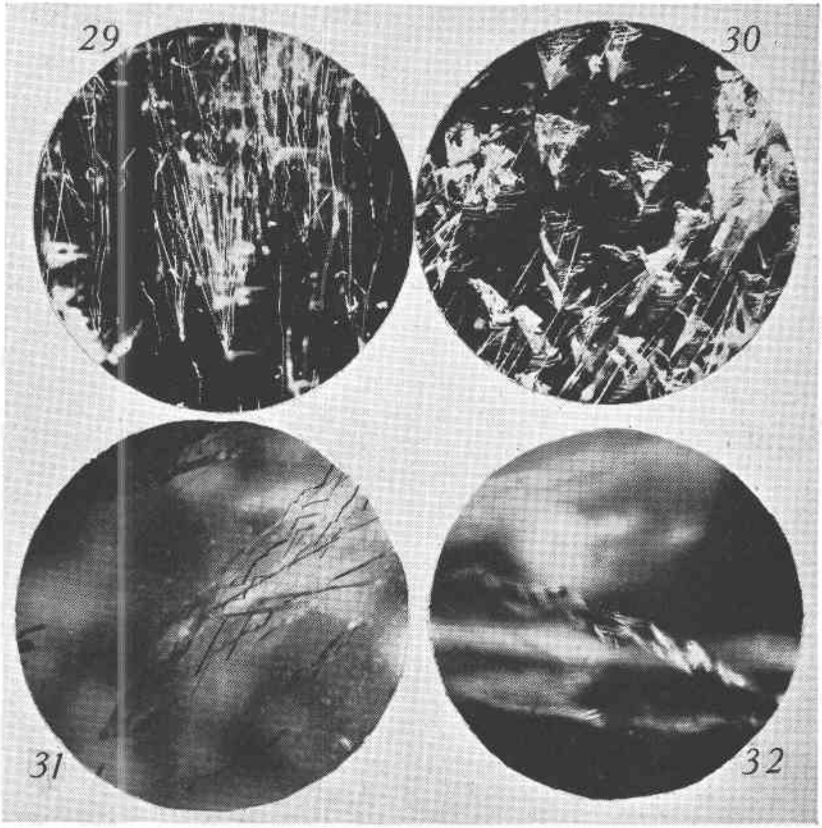
The "blue needles" are not reticular aggregates, such as shown by rutile inclusions in rose quartz, although the photographs give this illusion through projection on the same plane of V's at successive, and often equally spaced levels below the surface. "Blue needles" are not uncommon in quartz, but patience and alertness are necessary in hunting for them, since they become visible only in certain orientations in a powerful beam of light while immersed in oil: for example, the basal view shown in Fig. 28 showed the blue needles only when the beam of light struck a prism face above a major rhombohedral face *r* $(10\bar{1}1)$. Under a magnification of 30x, using a binocular microscope, the V is seen to include a number of finer needles radiating from the same point but not in the same plane. These finer needles are seen to be very narrow planes with a cross fiber structure: a single needle may be the intersection of two such planes. The fibers seem to be parallel to parting planes. Jutting out from the needles, at almost regular intervals, and more or less at right angles to the needles, are flat spear-heads: these seem to have a central line, which may have been a fiber, and a structure which appears to be made up of triangles or rhombs. They diffract light into the circular dots seen on the needles as "dew-drops" in the oil baths. These structures are hard to resolve, even under a magnification of 150×, but they seem nothing more than systems of linear parting planes. This interpretation is supported by examinations of etched wafers, where the etching solutions have penetrated the blue needles: only linear cavities bounded by planes parallel to parting directions (*r* $10\bar{1}1$) are seen (Figs. 29–32).

The Portugese word "chuva" meaning a fine rain is said to be applied to "white needles" with "small bubbles" giving the appearance of dew-drops along a thin fiber.[13]

Tyndall effect is a bluish smoky appearance of a beam of light passing through quartz, and probably due to the light scattering effect of extremely fine blue needles.

[12] This is obvious from the angle of 45° shown by the V's of fig. 27 as projected on the plane of the photograph.

[13] Willard, G. W., Raw quartz, its impurities and inspection: *Bell System Technical Jour.*, **22**, 338–361 (1943); *Mineral. Abstracts*, **9**, 83 (1944).



FIGS. 29–32. Photomicrographs by Dr. Thomas S. Stewart (magnification 7×, 7×, 22×, and 70× respectively), of a wafer which had been etched. It is assumed that the etching fluid has proceeded along "blue needles" which occurred in the quartz, because of the V-shaped clusters shown (Fig. 29). Fig. 30 shows the limbs of the V proceeding from the edges of the triangular-pyramidal etch pits on the opposite side of the wafer. Fig. 31 emphasizes how the solutions followed parting planes in the crystal. Fig. 32 shows the ribbon-like, linear character of the etched planes; under 150× magnification one can see optical twin boundaries on these planes!

Grading Quartz

To justify cutting, a quartz crystal must have some of its space free of twinning and detrimental imperfections. Its value will depend on how much of it is truly flawless. Efforts have therefore been made to grade quartz on the basis of its *usability:* defined as the estimated partial volume that might yield oscillator-plates.

Grading has heretofore been limited to the results obtained in the oil bath: on the basis of the amount of optical twinning found, and of such imperfections as is revealed in the intense beam of a spotlight. No effort has been made to grade quartz on the basis of the amount of electrical twinning present, since this would require etching of the crystals—and dealers object to anything being done to their quartz—in fact, the oil bath has already been too revealing.

Weight classification: Quartz is sold by weight, and is sorted into weights of 100 to 200 grams, 200 to 300 grams, 300 to 500 grams, etc. In the Bureau of Standards code, the numbers 1, 2, 3, etc. are used to designate the lower limit in a box of quartz.

Faced quartz refers to material having at least one identifiable prism or rhombohedral plane measuring approximately ¾ inch square or more. Boulders and defaced quartz are called unfaced quartz.

Optical quartz includes flawless material, 500 grams or more in weight, free of strain, but not necessarily free of electrical twinning. There are two grades of usability, 45–60% and 60–100%.

Oscillator quartz is graded[14] according to the estimated percentage of usable volume, as well as for the presence or absence of structures or defects that might limit its use for some purposes. Grade 1 includes flawless material. Grade 2 contains as defects only blue needles, color or Tyndall effect. Grade 3 shows in the eye-clear portions some parting, bubbles, and inclusions.

Grades 1 and 2 are graded further according to percentage of usability, with limits of 30–45%, 45–60%, and 60–100% (or roughly into more than a quarter, a half, or three-quarters usable). Grade 3 was classified into two grades of "usability" of 0–45% and 45–100%.

Inspection During Processing

There are many steps in the reduction of a quartz crystal into the tiny oscillator blanks: the quartz must first be sliced into wafers, or if large, into sections, and then into bars suitable for wafering. More than 95% of the quartz is useless or wasted in the processing, and the final oscillators may represent less than 1% of the volume of the quartz that one started with. Grading of raw quartz is necessary to be sure that the quartz has flawless areas that justify its processing. When the quartz has been cut into sections, bars, and wafers it is the practice to etch

[14] National Bureau of Standards, Optics Division (FJB:MLH, IV-w.Ipp-941-c) "Specifications for testing and grading crystalline quartz in conformity with the conditions imposed by the procurement, purchase, distribution, and use of the material as found by the Board of Economic Warfare, Metals Reserve Company, War Production Board, and the U. S. Army Signal Corps." Washington, D. C. 1943.

them, and reject hopelessly twinned areas. The most important stages of inspection, however, are those of the blanks prior to lapping and final finishing. If cut from bars they should be etched and examined for twinning; it is assumed that only untwinned blanks were diced from etched

FIGS. 33–35. Among the most important stages of inspection in processing are those of the blanks prior to lapping and prior to final finishing. Prior to lapping, blanks are inspected for internal imperfections (cracks, bubbles, inclusions) by immersing the blanks in oil and scanning them in an intense spot-light (Figs. 33–34). Prior to etching to frequency, lapped blanks are inspected in a powerful light for surface imperfections (scratches, chipped edges) (Fig. 35). (Blanks and drawings from a chart by Caroline Briggs and E. J. Korda (NAP).)

wafers. Prior to lapping, the blanks must be subjected to rigid inspection, while immersed in oil, in an intense spot light, for bubbles, cracks, inclusions, and other defects (Figs. 33–35). These defects, as well as twin boundaries, are areas of weakness, and the blanks are liable to crack in the laps—perhaps resulting in total loss of a lap load. Moreover, their rejection at this point eliminates lapping of useless material.

Lapping of blanks causes submicroscopic shattering of the surface to a depth of .25 to 0.5 microns. This shattered surface tends to disintegrate, and often, in the case of small, high frequency plates, results in total loss of activity. This deterioration, which has been dignified by the term "ageing," can be eliminated by dissolving the unstable, shattered surface. It is now, therefore, the practice to etch plates to their final frequency. Prior to such etching, the surface of the blanks must be examined in a strong spot-light to eliminate those that were scratched, since the etching solutions tend to attack those places, resulting in plates with lower activity.