SECONDARY DAUPHINÉ TWINNING IN QUARTZ

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ABSTRACT

Dauphiné twinning (so-called electrical twinning) can be produced artificially in quartz by: (1) cooling beta-quartz down through the 573° inversion point to alpha-quartz; (2) by rapid cooling of alpha-quartz down from roughly the 200° to 550° range; and (3) by the local application of high pressures at room temperature or higher. Secondary twinning produced below 573° is not accompanied by a permanent, geometrically defined, distortion of the crystal and is distinct from twin gliding produced by mechanical deformation. Additional types of secondary twins can be produced artificially by superposing Dauphiné twinning upon other types of twins already present.

The strategy of detwinning Dauphiné twinned oscillator-plates is based on heating the quartz over the inversion point to beta-quartz, in which Dauphiné twinning cannot exist by reason of symmetry, and controlling the cooling conditions so that the reinversion proceeds from one center rather than many. Natural Dauphiné twinned oscillator-plates homogenized by heating over 573° show a "memory" phenomena in which both the incidence and distribution of the secondary twinning which reappears on cooling are influenced by the original twinning. Secondary Dauphiné twinning in oscillator-plates usually exhibits a regular pattern dependent on the orientation and outward shape of the plate.

Both cracking and secondary twinning in quartz originate in cooling stresses and are primarily influenced by the rate of heating and cooling and by the size of the piece of quartz. While both effects are favored at 573°, neither is necessarily a concomitant of or indicative of inversion from beta-quartz. The conventional criteria for the distinction of natural alpha- and beta-quartz are criticized adversely in this connection. An added criterion of origin based on three-foldedness in areal distribution of natural Dauphiné and Brazil twinning is discussed.

INTRODUCTION

A very large proportion of the raw quartz available to the quartz oscillator-plate industry cannot be used because of the presence of twinning. A method of artificially removing twinning from oscillator-plates therefore would be of considerable practical importance and an investigation toward this end was undertaken. Unfortunately, more was learned about putting Dauphiné twinning into quartz than of taking it out. The

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results of the work, however, still are of interest, especially from their bearing on the use of quartz as a geologic thermometer, and are described beyond.

Types of Twinning in Quartz

The two most common twin laws in quartz, present in almost every crystal, are the Dauphiné and the Brazil. In the Dauphiné law, also known as orientational, 180-degree, or electrical twinning, the twinned parts are related as by a rotation of 180° around $Z=c$ and are of the same hand. Electrical twinning is objectionable in an oscillator-plate, for not only is the piezoelectric activity of the plate reduced or nullified due to the reversal of polarity of the polar $X=a$ axis in the two parts, but the 180° rotation alters the frequency-thickness constant and the temperature coefficient of frequency as well. In Brazil twinning the twinned parts are related as by reflection over $\{11\bar{2}0\}$ and are of unlike handedness. The composition surfaces usually are plane and parallel to $\{10\bar{1}1\}$ and $\{01\bar{1}1\}$ or, more commonly, to $\{10\bar{1}1\}$ alone, and the twinning is laminar. Brazil twinning is less objectionable than Dauphiné twinning because the oscillator-plate remains elastically homogeneous with identical frequency-thickness and temperature coefficients in the twinned parts, but the twin operation again reverses the polarity of the $X=a$ axes and hence reduces crystal activity. The term optical twinning as applied in the oscillator-plate industry refers to all twin laws that can be recognized by suitable optical tests; it comprises Brazil twinning primarily, since this is extremely common, but also includes the Japanese and all other known twin laws except the Dauphiné.

The strategy of removing Dauphiné twinning is based on heating the quartz over the alpha-beta inversion at 573°, at which point the twinned crystal becomes homogeneous, and then controlling the conditions of cooling so that the twinning does not reappear at lower temperatures. When a twinned crystal is inverted to a polymorph of higher symmetry the twinning will persist if the original twin operation does not become an operation to identity in the high symmetry phase. Furthermore, a twin that can be defined by a multiple operation such as a combined rotation and reflection may lose only one of the operations on inversion to the more symmetrical polymorph; conversely, an additional operation may be acquired when the inversion goes in the opposite direction. In the case of quartz, twins in which the crystal axes are not parallel or in which the twinned parts are of different handedness the twinning will persist over the 573° inversion point and cannot be removed. These include all known twins in quartz with the exception of the Dauphiné law. Dauphiné twinning can be removed from the quartz because the 180° rotation about the threefold [0001] axis of symmetry which defines the twin in ordinary quartz becomes an operation to identity in the sixfold [0001] axis of symmetry of beta-quartz. For the same reason, this law can be introduced into quartz together with multiple-operation twins which contain the Dauphiné twin operation. The introduced multiple-operation twins appear as relatively large areas of secondary Dauphiné twinning which locally cut across the pre-existing twin of another kind, thus "twinning the twin." Some examples are described on a later page.

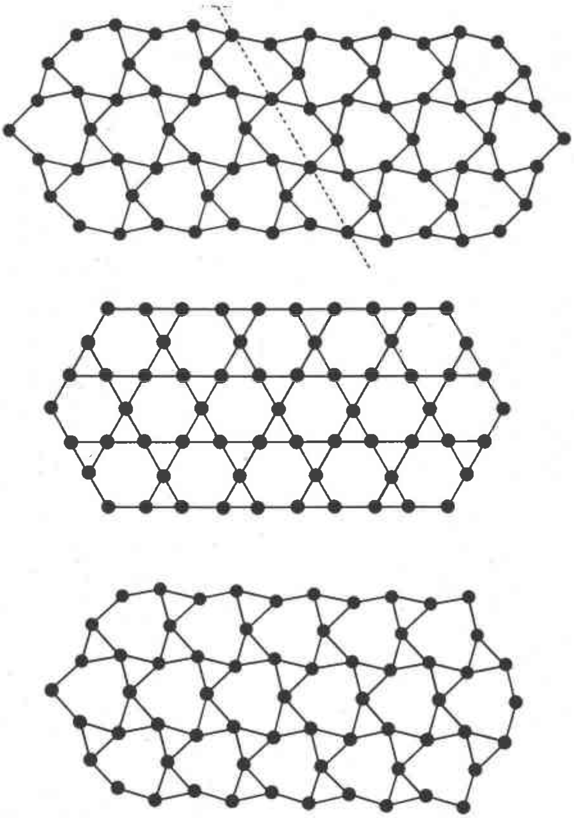

FIG. 1. Diagrams showing (top) the arrangement of Si atoms in (0001) planes of a Dauphiné twinned alpha-quartz crystal; (center) the same crystal heated above the inversion point to beta-quartz; (bottom) the same crystal cooled below 573° and re-inverted to untwinned alpha-quartz.

The relations between the structure of Dauphiné twinned alpha-quartz and of beta-quartz are shown in Fig. 1. The diagrams show the arrangement of the Si atoms in the [0001] planes. In the beta-quartz structure the threefold axis of alpha-quartz becomes sixfold and another set of horizontal twofold axes is added. The quartz remains right- or left-handed during inversion. As seen in the figure the crinkled rows of atoms straighten out in the beta-quartz structure. On reinversion to alpha-quartz the rows can crinkle, by chance, one way or the other. The two ways differ only by a rotation of 180° around $Z=c$. If both ways happen simultaneously in different parts of the crystal a Dauphiné twin results.

TWINNING PRODUCED BY INVERSION AT 573°

The amount of twinning produced by inversion at 573° is influenced by the rate of cooling through the inversion point, the size of the piece of quartz, and by the amount and distribution of Dauphiné twinning originally present. Generally speaking, slow cooling tends to increase the amount of twinning produced and to decrease the amount of cracking. Fast cooling has an opposite effect. With initially untwinned BT-cut oscillator-plates about 0.015 inch thick and 0.75 inch on edge it was found that all of the plates become twinned at cooling rates less than roughly 3° a minute in the range from 573° to 600°, and the loss due to cracking was negligible. Hundreds of plates were run simultaneously. At cooling rates of about 5° to 10° a minute from 40 to 70 per cent of the total number of plates came through the inversion point without twinning, provided that the plates were untwinned to begin with, and only 2 or 3 per cent of the plates were cracked. At faster cooling rates the incidence of twinning held about the same but the loss due to cracking became large. Inversion twinning always developed in plates of quartz over about 0.040 inch thick regardless of the cooling conditions. If the plates were originally twinned, that is, contained natural Dauphiné twinning, the per cent that came back without twinning was greatly reduced and even under optimum conditions amounted to less than 5 or 10 per cent. Theoretically it should make no difference whether the plates were twinned or not in the beginning since the twinning disappears anyway over 573°. Apparently strains are created and persist in the plate when the twinning is lost on heating through the inversion point, and these predispose the plates to twinning when cooling down below 573°. Annealing at temperatures as high as 1000° C is of no benefit. The irreversible beta-quartz to tridymite inversion at 870° is extremely sluggish and was not observed. It is also found that plates cut at −49° to the $Z=c$ axis come back, if untwinned, at the same minus angle as originally. This too is a memory phenomenon since theoretically the plate could reinvert as a single crystal in either the plus or minus positions, that is, in the relation of a Dauphiné twin. +49°, and + or −35° plates behave similarly.

Strain Patterns of Inversion Twinning. The distribution of Dauphiné inversion twinning in plates heated over 573° is quite interesting. Definite patterns are obtained which are somewhat dependent on the particular rate of cooling used. Some typical patterns are shown in Fig. 2. Rectangular, circular and triangular plates give distinct but related types of patterns. The twinning is distributed not only with respect to the geometrical outline of the plate but also to the crystallographic directions. Thus the triangular twinned areas seen in the fourth row of Fig. 2 taper in the direction of the X = *a* axis, and possibly also point toward a particular pole of this axis as well but it was not possible to prove this experimentally.

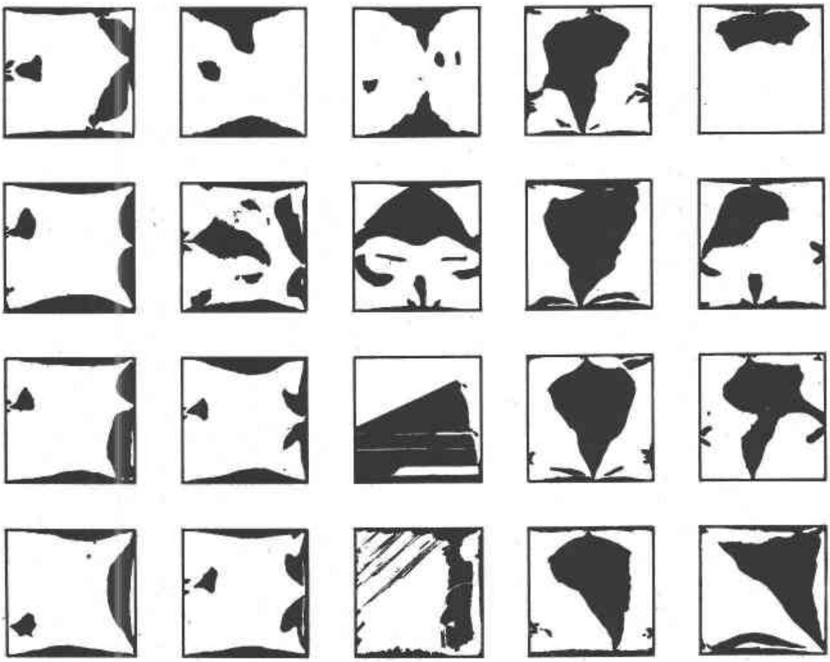

FIG. 2. BT-cut quartz oscillator-plates 0.75 inch on edge and 0.015 inch thick containing patterns of secondary Dauphiné twinning produced by inversion at 573°.

The patterns are seemingly related to elastic strains set up in the quartz during the heating and cooling cycle and give a sort of photoelastic or "twin elastic" analysis of the strains. The twinning is closely related to cracks in the plates, as shown in Fig. 3. In one experiment the twin pattern resembled, quite appropriately, the map of South America. If the plates are loaded artificially so that they are under imposed outside stress other types of patterns are produced. Occasionally patterns are obtained that consist of straight-sided parallel laths or rectangles which simulate Brazil twins. The margins of the twins do not cut perpendicularly through the quartz in BT plates but usually dip inward at a shallow angle so that the shape of the pattern can be changed considerably by lapping or deeply etching the plate. Large irregular pieces of quartz do not give regular patterns of inversion twinning.

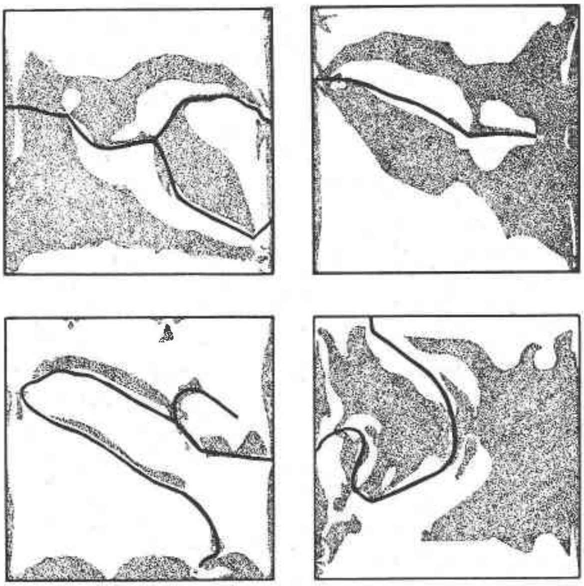

FIG. 3. BT-cut quartz oscillator-plates heated through 573° and showing the relation of cracks to secondary Dauphiné twinning.

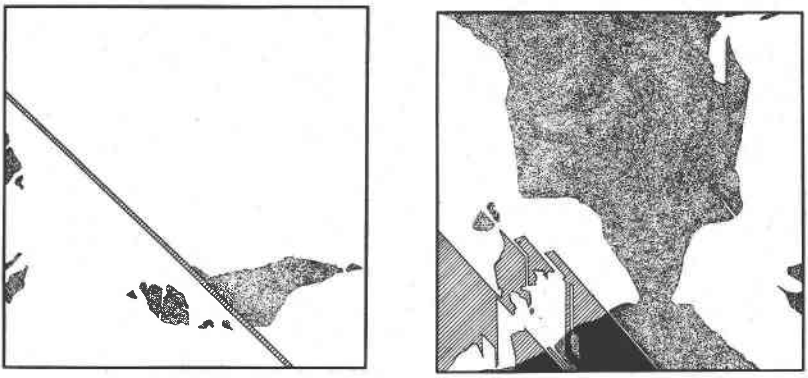

FIG. 4. BT-cut quartz oscillator-plates containing both secondary Dauphiné twinning (stippled) and original Brazil twinning (ruled). Twinning on the "Combined law" (black) is generated where the secondary Dauphiné twin cuts across the Brazil twins.

Combined Twin Law. Quartz oscillator-plates in which secondary Dauphiné twinning has been superposed upon original Brazil twins are shown in Fig. 4. The plates were cut from quartz containing Brazil twins only, and the secondary twinning was introduced by inversion at 573°. The Dauphiné twin boundary usually is deflected somewhat or may be stopped at the point where it enters the Brazil twin (Fig. 4). The "twinned twin" can be described as comprising a combination of reflection over $\{11\bar{2}0\}$ and a rotation of 180° around [0001]. This law, for which the name "Combined twin" is proposed was described largely on theoretical grounds by W. J. Lewis.[1] Lewis described what seems to be a natural example of the twin in amethyst. F. Leydolt[2] earlier described etched sections of quartz which apparently exhibit the same type of twinning. The twin law, however, is not mentioned in the 6th edition of Dana's *System* or in Hintze's *Handbuch.* Recently, Captain C. F. Booth and associates of the Post Office Research Station, Dollis Hill, London, found a few natural examples among several thousand sawn and etched quartz crystals. A description of the twin together with excellent photomicrographs of twinned etch patterns has been published.[3] Several examples have also been found by Mr. G. W. Willard of the Bell Telephone Laboratories, in the course of a study[4] of twinned quartz, and a fourth natural instance has been brought to the writer's attention by Mr. S. G. Gordon, of the Academy of Natural Sciences, Philadelphia. In the natural instances the twin has formed by growth, and a superposed secondary twinning mechanism of the kind described above is not involved. Over 573° Combined twins become identical with Brazil twins by loss of the 180° rotation.

TWINNING PRODUCED BY RAPID COOLING FROM THE 200° TO 550° RANGE

Hitherto it has been thought that Dauphiné twinning could be produced artificially by heat only at the 573° inversion point. It is found, however, that typical "inversion twinning" can be produced by rapidly cooling or quenching thin oscillator-plates that have been heated to temperatures in the range from 200° to 550°. The incidence of twinning increases with temperature. At 200° only a few per cent of the total number of plates run became twinned and no twinning has been observed below 200°. The hot plates were quenched by dropping on a wet towel or into cold water. Cooling the plates for a few minutes in air before quenching

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[1] Lewis, W. J., *A Treatise on Crystallography,* London **(1899)**, pp. 522.

[2] Leydolt, F., *Ak. Wiss. Wien, Ber.,* **15,** 59 (1855).

[3] Booth, C. F., and others, *Post Office Eng. Dept.,* Radio Rpt. **449,** London (1939); also *P. O. Elect. Eng. J.,* **31,** 1 (1939).

[4] Willard, G. W., *Bell System Tech. J.,* **23,** 11 (1944).

tends to increase the amount of twinning. Under these conditions twinning is obtained in most or all of the plates that have been heated over about 350°. The quenching cracks the plates, especially at higher temperatures and when using cold water. The twinning usually develops in the uncracked areas and very closely cracked plates do not exhibit twinning—presumably due to the release of strains. Relatively small amounts of twinning are produced if the plates are removed from the oven and are not quenched but are allowed to cool rapidly in air. In no instance has twinning been observed in plates cooled slowly from temperatures below 550°.

The secondary twinning produced below 550° usually is rather patchy or forms small areas or strips along the sides of the plates. Some plates become spotted with rounded twinned areas up to 1 mm across. Plates quenched from 450° to 550° often show a peculiar clover-leaf arrangement of twinned areas as shown in Fig. 5. This arrangement suggests the regular geometrical twinned pattern produced at room temperature under high pressure, described beyond.

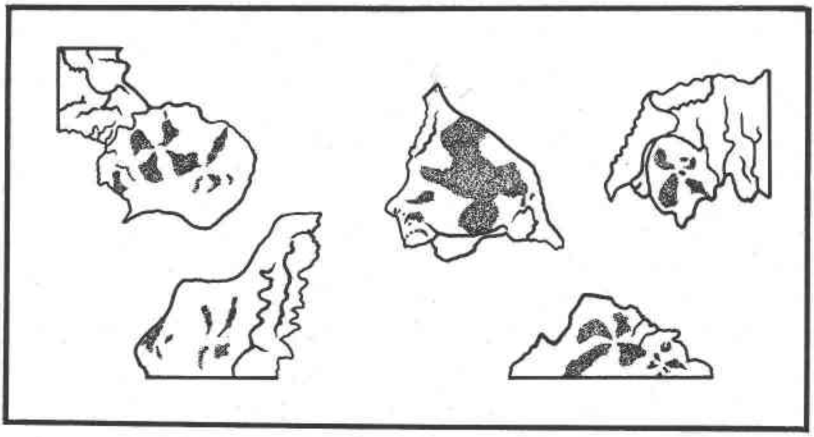

FIG. 5. Clover-leaf patterns of secondary Dauphiné twinning in cracked and broken BT plates quenched from 400°C.

The experimental observations indicate that low temperature twinning is brought about by cooling stresses in the quartz. The atomic mechanism by which the twinning is produced evidently differs from that of ordinary mechanical twinning or twin gliding since there is no evidence of permanent deformation of the quartz. Low temperature secondary twinning is obviously related to the Dauphiné twinning produced artificially by pressure at ordinary temperatures, for which a mechanism based on mechanical rotation of the SiO_4 tetrahedra has been proposed by Schubnikow and Zinserling.[5] It is interesting to speculate that possibly some of the natural Dauphiné twinning found in large crystals of alpha-quartz is produced by strains set up as the crystals cool from their original temperature of formation. In most cases, however, the natural twinning clearly is original, as shown by the discontinuity of striations and other surface growth features across the twin boundaries, by reentrant angles, such as on the *x* faces, and by the control exerted by the twinning over the distribution of color in amethystine and smoky quartz.

CRACKING IN QUARTZ

The development of cracks in quartz depends primarily on the size of the piece of quartz and on the rate of heating and cooling. Quartz, unlike silica glass, has relatively large coefficients of linear thermal expansion. The cracking is due to powerful stresses set up by the thermal gradient existing during heating and cooling, and to a volume change of about 0.86 per cent in passing through the inversion point. The stresses resulting from non-uniform distribution of temperature also generate surface changes at the ends of the electric $X = a$ axes equal to those produced by an equivalent mechanically applied stress. This is the origin of all so-called pyroelectricity in quartz; examples of true pyroelectricity in crystals are not known, with the possible exception of tourmaline.[6]

Plates of quartz 0.75 inch square and 0.009 to 0.015 inch thick can be heated to 700° to 1000° C at rates at least as high as 7° a minute without cracking more than a few per cent of the pieces. With plates 0.050 inch thick most or all become cracked at rates over at least 5° per minute, while only a small per cent is cracked at rates of 1° a minute or less. Larger pieces of quartz are more sensitive. Masses of quartz 2 or 3 inches thick almost invariably shatter on a minute scale when heated at rates of 0.5° a minute (the slowest rate tried) to temperatures over 573°, and pronounced cracking is obtained in pieces down to an inch or so in size. Marked cracking also is produced in pieces of this size when heated rapidly to temperatures in the range from 250° to 350°. On the other hand, if the rate of heating and cooling is reduced to 0.5° or so a minute and the quartz is soaked at constant temperature for an hour or so every 30° to 40° interval over this heating and cooling cycle, the cracking can be entirely eliminated. This method has been used to heat large smoky quartz crystals to *ca.* 325° for the purpose of decolorization.

Quartz that initially contains cracks, bubbles or inclusions is found to

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[5] Schubnikow, A., and Zinserling, K., *Zeit. Krist.*, **83**, 243 (1932); Zinserling, K. and Schubnikow, A., *Zeit. Krist.*, **85**, 454 (1933).

[6] Wooster, W. A., *Textbook on Crystal Physics*, London **(1938)**, p. 226.

crack readily on heating. Cracks that develop in thin plates heated over the inversion point are curved and irregular (Fig. 3); and there is a tendency for many of the cracks to stop short of the margin of the plate. Thin plates heated to 250° to 450° and quenched in water often develop intricate fingerprint-like patterns of cracks. The cracks sometimes have a narrow margin of secondary Dauphiné twinning, or are obviously related in distribution to the twinning (Fig. 3). The twinning usually is near the crack but not directly on it, or the twinning appears in areas of the plate that are not minutely cracked. The latter relation is especially marked in plates that have been twinned at low temperatures. The cracking apparently releases the strains that cause the twinning in the first place. It is of interest in this connection that Mügge[7] found thin quartz plates momentarily warp as much as 3° when passing through the inversion point.

TWINNING PRODUCED BY HIGH PRESSURES AT ROOM TEMPERATURE

Schubnikow and Zinserling[5] found that Dauphiné twinning could be produced at room temperatures by local application of pressures over about 1000 kilograms per sq. cm. The pressure was applied over a period of a few days. Small geometrically shaped twinned areas develop at the point of pressure. The shape of the areas varies with the orientation of the quartz, as shown in Fig. 6 (taken from the original publication). Further work along these lines has been done in connection with the present study, using a simple lever arm press or a vise to apply pressure to the quartz through a $\frac{3}{8}$ inch diameter steel ball bearing. In one experiment twinning was produced in a $\{11\bar{2}0\}$ section with 330 pounds ball pressure,

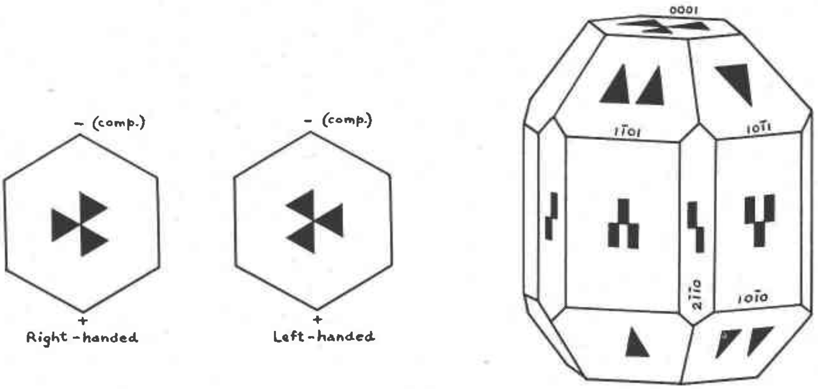

FIG. 6. Geometrical patterns of secondary Dauphiné twinning produced by local pressure at room temperature (after Schubnikow and Zinserling).

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[7] Mügge, O., *Jb. Min., Festband*, 181 (1907).

and in a BT section with 610 pounds ball pressure. The twinned areas were only a millimeter across. The surface of the quartz must be lapped down slightly and then etched in order to reveal the twinning.

QUARTZ AS A GEOLOGIC THERMOMETER

The criteria commonly applied to the distinction of natural quartz formed under 573° (alpha or low quartz) from quartz formed above 573° (beta or high quartz) are: (1) the presence of general forms proper to the point groups of alpha- or beta-quartz. In the absence of general forms, prismatic development and an unequal development of the terminal rhombohedrons is considered indicative of the alpha form while an equant pyramidal habit is thought typical of beta-quartz; (2) the character of the Dauphiné twinning, which has been thought to be usually regular and sharply marked in alpha-quartz, and to be irregular, small, patchy, and without relation to the external form of the crystal in material that has cooled through the 573° inversion point; (3) intergrowths of right- and left-handed quartz (Brazil twins) are said to be more frequent and regular in boundary line in the alpha than in the beta form; (4) alpha-quartz formed by inversion from beta-quartz is relatively cracked and shattered and tends to crumble when etched in hydrofluoric acid. These criteria were suggested by Wright and Larsen[8] and earlier in part by Mügge.[7] Wright and Larsen point out that the criteria (with the exception of those based on the occurrence of general forms) are not rigorous but contain an element of probability. Later workers have applied the criteria with some confidence. The present experimental observations, in the writer's opinion, greatly increase the uncertainty attending their application.

Secondary Dauphiné twinning is not peculiar to or necessarily indicative of inversion at 573°. It can be produced thermally at least as low as 200° and can be induced mechanically at room temperature. In most instances the boundaries of the secondary twinning are irregular and blotchy, as pointed out by Wright and Larsen, but these features are found to be produced both above and below 573°. Further, straight boundaries also occur in secondary twins (cf. Fig. 2). Some secondary Dauphiné twinning simulates the regular characteristics of Brazil twinning. As a matter of fact both the irregular patchy type and the straight-sided sharply marked type commonly occur in natural undoubted alpha-quartz. A graphic type of natural Dauphiné twinning in what is presumably alpha-quartz has been figured by Leydolt.[2]

Cracking is largely a matter of the rate of heating or cooling. It seems probable under geologic rates of temperature change that quite large pieces of quartz could come through the inversion point without marked

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[8] Wright, F. E., and Larsen, E. S., *Amer. J. Sci.*, **27**, 421 (1909).

cracking. The mechanical history of the quartz, especially when massive or embedded, as in the case of most pegmatitic quartz and the quartz grains of igneous rocks, no doubt is an added and important factor. Uncracked oscillator-plates returned from 1000° do not crumble when deeply etched in acid; the plates are clear and flawless and, if nearly or entirely free from twinning, stand up under oscillation.

The occurrence of general forms is primafacie evidence of origin, but general forms do not necessarily occur on every euhedral quartz crystal and in fact have not yet been observed on so-thought beta-quartz. While an unequal development of the terminal rhombohedrons would be expected for structural reasons to be characteristic of alpha-quartz, it may be noted that crystals with an equant development of the rhombohedrons and without prism faces—the so-called beta-quartz habit—are not uncommon in the alpha variety. This habit is typical of certain low temperature iron ore deposits, such as Cumberland, England, and Antwerp, New York, and has been suggested as a criterion of deposition from iron-rich solutions.

Criteria based on the relative frequency of Brazil twinning in alpha- and beta-quartz are not backed by independent rigorous evidence and were originally derived largely from theoretical considerations. Examination of literally thousands of etched sections cut from euhedral crystals or alpha-quartz, in most cases proven as such by the presence of general forms, clearly indicate that the relative development of Brazil and Dauphiné twinning varies widely, as does the individual percentage of these types of twinning. The type of occurrence seems to be a controlling factor.

Threefoldedness in Distribution of Twinning as a Criterion of Origin. A new line of evidence that appears to be of some value in distinguishing alpha- from beta-quartz concerns the symmetry of distribution of the twinning. Basal sections of material known to be originally alpha-quartz often show when etched a threefold distribution of the Dauphiné or Brazil twinning. Some typical examples are shown in Figs. 7 and 8. The instances of Brazil twinning are rough copies of figures given in the literature[9]; other examples of threefold Dauphiné twinning have been figured by Leydolt.[2] Threefoldedness in the distribution of Brazil twinning seems to indicate that the quartz did not crystallize over 573°, since this law has an equivalent in beta-quartz and would then have a sixfold areal distribution, if any. The threefoldedness could not indicate whether an initially alpha crystal was later heated over 573° or not since Brazil twinning is not affected at the inversion point. Similarly, threefoldedness in the distribution of Dauphiné twinning indicates that the quartz origi-

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[9] Cf. Descloizeaux, A., *Ann. Chim. et Phys.* (3), **45**, 129 (1855); *Mem. Ac. Sci.*, **15**, 404 (1858); Groth, P., *Zeit. Krist.*, **1**, 297 (1877); Tutton, A. E. H., *Crystals*, London (**1911**), p. 222; Leydolt, F., *op. cit.*

nally crystallized in the alpha range of temperature, since secondary Dauphiné twinning resulting from inversion from the hexagonal beta phase presumably would have a sixfold distribution, if any. It is question-

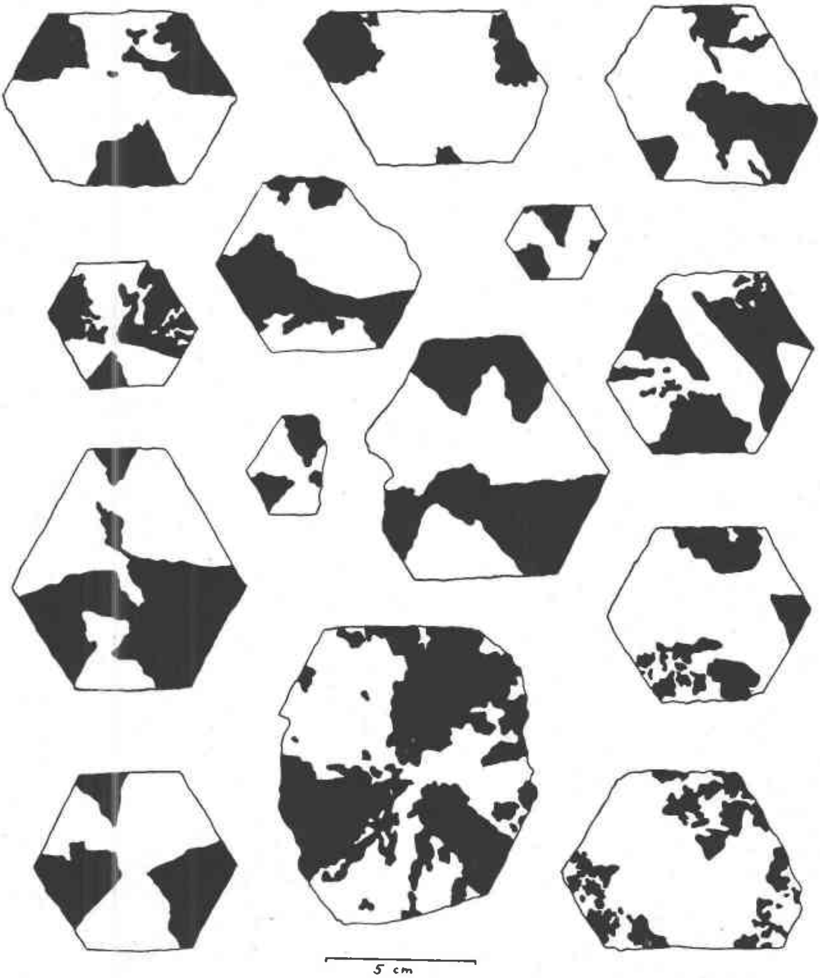

FIG. 7. Sawn and etched basal sections of alpha-quartz showing a threefold distribution of natural Dauphiné twinning.

able whether threefoldedness of distribution can be used as evidence that an initially alpha crystal subsequently was not heated over 573° since, as has been noted, inverted quartz contains residual strains or areally distributed defects which might predispose the crystal to a three-folded distribution of secondary twinning resulting from inversion.

Brazil and Dauphiné twinning are frequently found to be closely related in distribution to the external morphology. The Brazil twins often correspond in size and distribution to the terminal $(10\bar{1}1)$ faces and may be entirely restricted to the growth loci of these faces. The Dauphiné twins apear to be related to the modifying trigonal trapezohedral faces. These relations seem to be genetic. If this is the case, the symmetry of distribution reflects definitive characters of the trigonal trapezohedral crystal class and hence is rigorous evidence of crystal symmetry.

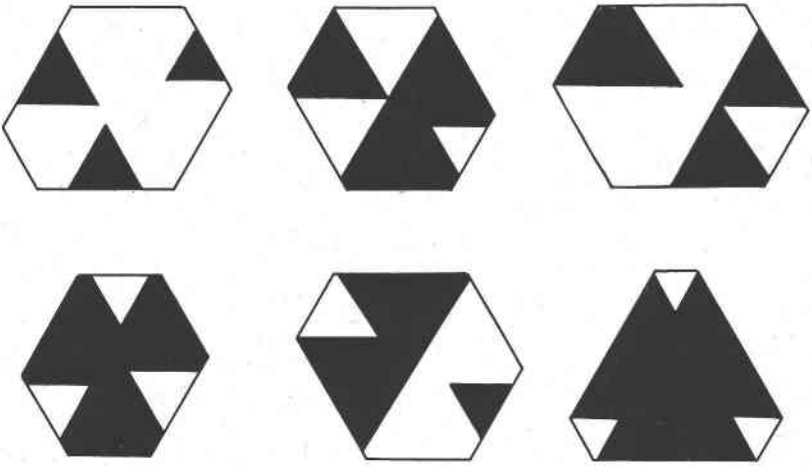

Fig. 8. Threefold distribution of Brazil twinning (black) in basal sections of amethystine alpha-quartz (rough copies of figures in the literature). The twinned areas in all instances are subjacent to the faces of $(10\bar{1}1)$.

Threefoldedness in the areal distribution of various fortuitous characteristics of crystals such as color or inclusions similarly could be used as evidence of origin. A threefold distribution of color is quite common in amethyst and is occasionally observed in smoky quartz. In these instances the color ordinarily is restricted to the growth segments beneath the terminal $(10\bar{1}1)$ faces and is absent from the face loci of $(10\bar{1}0)$ and $(01\bar{1}1)$. Minor amounts of foreign elements held in substitutional solid solution or otherwise in crystals often are areally distributed with respect to the external morphology and this, too, is indicative of crystal symmetry; numerous examples have been described by Frondel, Newhouse and Jarrell.[10] It is also possible that criteria more subtle than areal distribution or symmetry may be found to aid in the problem of distinguishing alpha- and beta-quartz. In particular it may be profitable to investigate order-disorder and defect-structure phenomena and mosaic structure.

[10] Frondel, C., Newhouse, W. H., and Jarrell, R. F., *Am. Mineral.*, **27**, 726 (1942).