

FRACTOGRAPHY AS A MINERALOGICAL TECHNIQUE*

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SUMMARY

Fractography, the special microscope technique developed during the past decade in the field of metallurgy, is applied to a preliminary study of several minerals for purposes of introducing fractography to mineralogists as a possibly useful petrographic method.

First, a general classification of fracture patterns is established to separate the markings relating to structures intrinsic to the crystal (Type I) from those caused by local resolutions of the transient fracturing stress (Type II). Examples are shown using metals and synthetic chemical crystals; and these in turn provide a further subclassification of the Type I patterns—which are the patterns of greatest significance—into (1) Type Ia, representing the crystallographic directionalism of the lattice structure, (2) Type Ib, expressing crystal imperfection following from failure to attain an ideal lattice structure, and (3) Type Ic, disclosing the presence of extraneous phases.

On the basis of these categories, a number of minerals are studied fractographically, including plagioclase, orthoclase, muscovite, fluorite, dolomite, twinned calcite, and optically pure calcite. The patterns are found to have two aspects of broad importance: (1) petrographic and (2) micellar.

With regard to the first, the following phenomena are illustrated: (*a*) twinning, (*b*) parting on twin planes, (*c*) perfect cleavage, (*d*) intersecting primary and secondary cleavages, (*e*) fine superficial striae, possibly related to twinning, (*f*) occluded interlamellar phases in muscovite, (*g*) inclusions, both solid and fluid, and (*h*) markings of unknown origin.

As for the micellar aspect, reference is made to the current widespread discussions of theories attempting to reconcile the vast differences between “ideal” and “real” crystals, and specifically to the recent “micellar theory” which proposes a universal subdivisional nature for the solid state inherited from a micellar condition in the liquid. Fractographs of the minerals, as of the metals, prove to be rich in evidence for such a mosaic or micellar constitution; and some elaborate imperfection patterns are presented.

INTRODUCTION

During the past decade there has been developed in the field of metallurgy a specialized micrographic technique now known as “fractography” (1, 2, 3). As the name implies, this technique concerns the study of detail on fracture surfaces. Because mineralogy has concerned itself with the appearance of fractures virtually since that science began, one might wonder whether there is anything further to be gained from it.

In the field of metallurgy, the situation is somewhat different, for the fractures of most metals have a roughness and a multiplicity of minute crystals which make direct microscopic observation difficult and forbidding. De Réaumur (4) made some historically interesting sketches of

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the fractures of iron and steel in 1722, based upon microscopic examination which probably followed contemporary mineralogical practice; nevertheless, after Martens' work (5) in 1887 was abandoned in favor of the method of direct study of fractures, what has now become conventional metallography—the cutting, polishing, and etching of solid sections and their study by reflected light.

Glancing briefly at the parallel history of mineralogy (6), one finds Henry Clifton Sorby in 1849 publishing in his "Calcareous Grit of Scarborough" the first work on thin sections of minerals. His attention had been attracted to a study by Williamson on thin slides of fossil wood. Sorby wrote again on "Slaty Cleavage" in 1851; and in 1857 he presented a paper on "The Microscopic Structure of Crystals" before the Geological Society of London. For many years Sorby's work received little attention, although today the thin-section technique is the rampart of petrography.

An attempt by Sorby to apply the thin-section technique to metals failed, of course; but this led to his development of the polish-etch method of modern metallography. The two fields of mineralogy and metallurgy thereafter separated, so far as techniques for discerning structure and constitution were concerned.

In the development of fractography, however, attention has again been turned toward the detail to be found on cleavage and fracture surfaces. Certain technical improvements in microscope construction have appeared since Martens' time; and manipulations have recently been found which enable the direct observation of surfaces, almost regardless of their roughness. Furthermore, the technique as it is most fruitfully applied concerns high magnifications almost exclusively—from one- or two-hundred diameters up to the limits of the lens system. This change in scale of observation carries the work into categories not touched by the early workers.

Consequently, the aim of the present writing is to present to mineralogists a brief and generalized treatment of various exemplary metal and mineral systems for purposes of calling the attention of that profession to possible contributions which might be made by fractography applied to minerals. Some of the work to be shown has undoubtedly been observed before by certain mineralogists, and probably more effectively. Nevertheless, some of its aspects are new.

In addition, because of the rapidly mounting interest in theories for subtle substructures in the solid state—the so-called mosaic (7) and micellar (8)* theories, for example—a review of details found on nascent fracture surfaces in all solids is well warranted. As this paper will clearly

* See Appendix for an outline of the micellar theory.

illustrate, fractography discloses impressive observations of a subdivisional architecture within the crystal, observations which can have much to do with the formulation of an understanding for certain characteristics of solids which at present have no satisfactory explanation. One is here reminded of Haüy, the discoverer of the Law of Rational Indices and the founder of modern crystallography, who a century and a half ago described crystals as physical composites of a "crystal molecule" (9) and sketched the gross structures as a brickwork of submicroscopic perfect crystallites. His concept was driven under by the impetus of atomic theory, the space lattice, and particularly *x*-ray diffraction; nevertheless, there has redeveloped in the past quarter century some rapidly mounting evidence that such subdivisional structure does exist between the molecule or unit cell and the single crystal.

This text will accordingly divide its attention between: (1) evidences in fractographs having promise from a petrographic or crystallographic standpoint, and (2) evidences for subdivisional architecture having significance for theories of the solid state.

EXPERIMENTAL METHOD

A number of papers have already appeared on fractography; and to avoid some repetition here, Ref. No. 3 has been listed in the bibliography. In that paper a complete bibliography of fractographic studies through 1948 can be found, and these in turn can be used to supply details of the technique as developed to that date. The first three papers listed in the present bibliography also contain descriptions of the two types of fractographic stages used thus far. These descriptions are not necessary to repeat here because the fractographic stage is little other than a simplified orienting mechanism, whose general principles are well known to mineralogists.

In brief outline, an orienting mechanism holds a fractured chip in a clamp or in a plasticene cup, with the fracture facet roughly perpendicular to the microscope axis. A lens providing a magnification in the order of 100 \times is brought toward the fracture face until a rough focus is obtained. The stage is then rotated and shifted until some portion of the fracture field attains good perpendicularity and yields a usefully flat field. Such an area will announce its arrival in the field by a flash of light. A stronger lens can then be inserted, if desired, the focus refined, and the field explored.

Magnifications up to the limit of the microscope are readily used, at least on opaque bodies; and magnifications greater than 100 \times are generally recommended.

Illumination by any of the common processes is applicable, including:

(a) vertical illumination, (b) oblique illumination, (c) dark-field, (d) phase-contrast, (e) sensitive tint, and (f) polarized light; however, the procedure for the work as developed to date, and as used in this paper, comprises vertical illumination (inverted metallurgical microscope) *slightly obliqued to provide surface contrast*. The importance of obliquing, particularly for photography, cannot be overemphasized.

Specimens include any solid whose fracture surface can be isolated for study. If promontories interfere, they can be removed by delicate hand grinding operations; but often the promontories themselves have on them informative structures. While the facet can be polished or etched, or treated much as desired, the primary basis for fractography lies in the nascent, unetched, and untouched fracture surface.

PATTERN TYPES

Before considering the fracture patterns, two broad types of markings must be defined:

Type I: Patterns developed by factors intrinsic to the solid.

Type II: Patterns imposed by factors extrinsic to the solid.

In the first, Type I, the path of fracture follows a traverse influenced by, and hence expressing, the structural forms and directional weaknesses within the solid itself, and therefore reveals directly the architecture of the specimen. This classification, Type I, contains by far the more useful information; and because of its prolific registrations, a number of subclasses can subsequently be named.

For example, in Fig. 1 there are reproduced two fractographs taken of cleavage surfaces in cast bismuth metal. The upper, representing cleavage on the basal (0001) plane, discloses two broad bands which are shaded gray by the oblique illumination and are known to be twins on $\{10\bar{1}4\}$. Also, on close scrutiny of this fractograph one will perhaps discern, even in the reproduction, faint parallel lines of "striae" running along three sets of directions at 60° to one another. While the origin of these has not been positively identified (10, 11, 12), the point is readily allowed that the two groups of phenomena—twins and striae—have created a pattern on the fracture traverse because of a fundamental crystallographic directionalism. *Type Ia* can therefore be named as a subclass referring to *deflections in the fracture traverse caused by the directionalism of the atomic lattice structure*.

In the lower fractograph of Fig. 1, cleavage on an inclined plane of $\{10\bar{1}1\}$ form is depicted. Contrast to the basal cleavage pattern is marked. Registrations of the twin bands again appear, but much altered in structure and, of course, in respective angularities, since the planar intersection is now $\{10\bar{1}4\}$ upon $(10\bar{1}1)$. One can orient this pattern by recogniz-

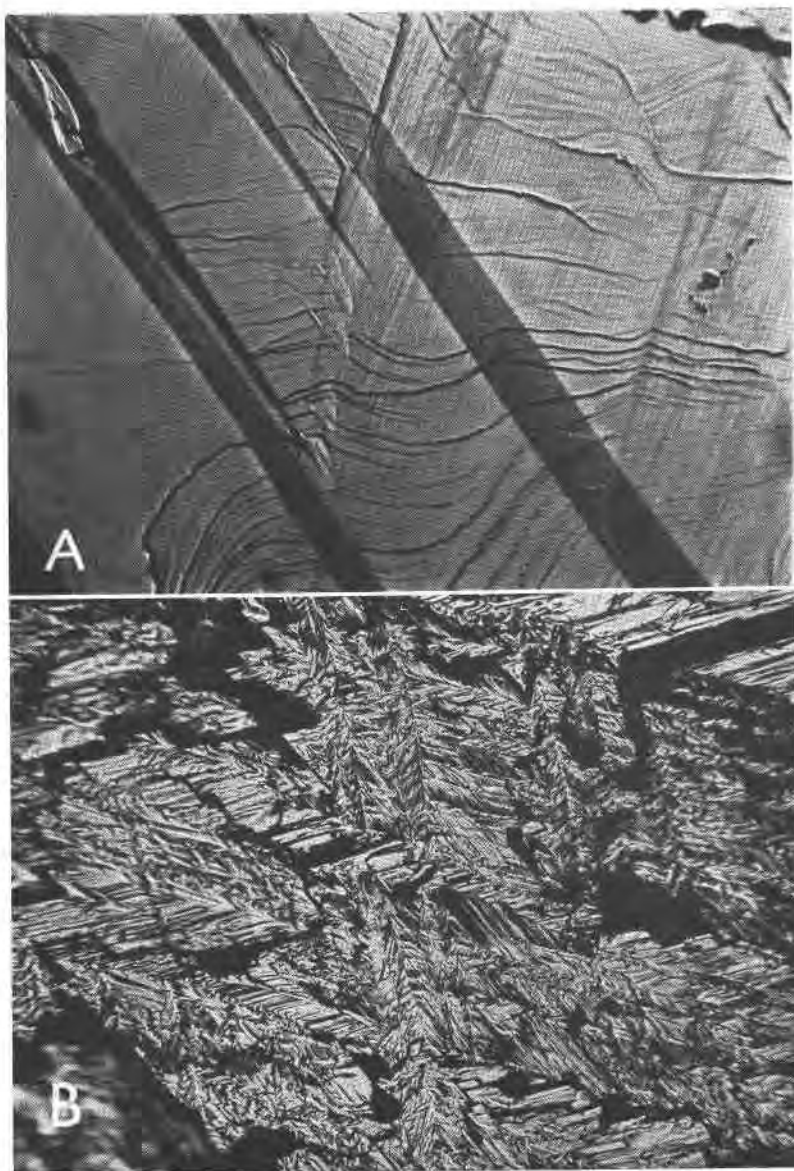


FIG. 1. Fractographs of cleavage in commercially pure cast bismuth illustrating Types Ia and Ib patterns for a homogeneous constitution. (Above) Basal cleavage on (0001) at 550 \times . (Below) Secondary cleavage on (10 $\bar{1}$ 1) at 170 \times .

ing the small sharp profiles as (0001) cleavages intersecting the (10 $\bar{1}1$) plane of the fractograph. The herringbone pattern then displays the twin by the small laminar basal cleavages now reoriented along the twinned layer.

Since it is not the purpose of the present writing to describe the crystallographic details of deformation and fracture in bismuth, discussion of this second fractograph will turn its attention instead toward the remarkable substructural detail. Here is direct evidence of an impressive kind that this single crystal of bismuth (the grain boundary lies far outside the photograph) comprises an elaborate substructure of a mosaic or micellar type (8), showing here particularly as laminae. Because this observation will become much fortified by later evidence, the feature can be at least tentatively accepted as warranting a *Type Ib* subclass for patterns expressing *directionalism resulting from imperfection in the development of the ideal lattice structure*. This will include in turn a broad category extending from the postulated micellar structure up through such disturbances as dendrites and grain boundaries. One will observe, however, that both subclasses Ia and Ib refer specifically to physical nonuniformities within a single homogeneous phase. These are structural features which can concern the presence of a second phase, but do not require it.

On the other hand, turning to Fig. 2, one finds strongly marked fracture characteristics which are again clearly intrinsic to the crystal, but are specific registrations of a second phase. The specimens here are molybdenum metal, vacuum-cast by an electric arc process (13, 14). In the upper fractograph there appear featherlike forms, now known to represent molybdenum carbide; and their position is characteristically intergranular.

In the lower fractograph, similar "carbide feathers" are again evident in the upper portion of the field; and encroaching upon this field are dendritic growths of molybdenum oxide, also intergranular. The chemical reaction of these two compounds under the high-temperature conditions of melting yields escapable carbon oxide gas; and one can observe the disappearance of the carbide toward the lower portions of the field. These two fractographs display the phenomena, incidentally, which control the forgeability of this metal.

Molybdenum displaying the upper fracture pattern is forgeable. That displaying oxide is nonforgeable and must be remelted.

Consequently, the fractographs in Fig. 2 clearly define a third subclassification under Type I. The pattern is intrinsic to the system, but *refers to the intervention of a heterogeneous constitution* and will be defined as *Type Ic*.



FIG. 2. Fractographs of cast molybdenum metal illustrating Type Ic patterns for heterogeneous constitution. (Above) Featherlike intergranular structures expressing carbide. 440 \times . (Below) Dendritic growths of intergranular oxide encroaching upon featherlike carbides as shown above. 320 \times .

Proceeding now to Fig. 3, one observes a pattern which is distinctive both in appearance and in cause. This is the Type II classification, resulting from factors extrinsic to the specimen—specifically stress. While subclassifications can be added to this category likewise, the present discussion will limit itself to a broad consideration of stress as the extrinsic factor.

Thus, the upper fractograph illustrates the typical pattern relating to the traverse of fracture through a matrix which imposes none of its own directionalism upon that traverse. The pattern is essentially a stress pattern, revealing the undulations and nodes of the shattering wave; and the path is similar for vitreous or amorphous bodies and for crystals exhibiting minor directionalism. Here the specimen is a synthetic crystal of ammonium dihydrogen phosphate, whose tetragonal structure perhaps should be expected to show Type I effects. In fact, it does to some extent, as is shown elsewhere (15); but there also occurs much Type II traverse, of which Fig. 3A is an example.

In the lower fractograph of Fig. 3 another fracture of this material is shown, photographed with dark-field illumination. The pattern is again predominantly Type II, although an elaboration occurs which is somewhat suggestive of lamellar Type Ib effects.

In summary, these three sets of fractographs convey the principal generalizations to be understood in interpreting the following fractographs of minerals. All of the Type I subclasses are generic to the study of the minerals to be discussed. Type II patterns are of no particular interest for the work at hand, and are to be distinguished only for purposes of their separation from the more informative markings.

In closing the discussion of pattern types, a particularly interesting example of the fractographic technique is given in Fig. 4. The specimen is isometric alpha iron (ferrite) which has been rendered brittle by absorption of hydrogen gas during treatment in sulfuric acid. The sample is polycrystalline, typical of irons and steels, whose grains are much smaller than those crystals commonly explored in mineralogy, and which therefore provide a problem for which fractography is specifically designed. The fracture of the specimen presented a typical rough appearance; but a single cleavage facet was selected and photographed. The corresponding half of the specimen was next oriented on the stage and explored until the matching facet was found. It, too, was photographed, at the same orientation and magnification. The pair of fractographs in Fig. 4 then provide an "obverse" and "reverse" view of the fracture traverse through this particular grain.

In these two matching patterns one will first note the rectilinearity, which immediately identifies it as being predominantly Type I. The

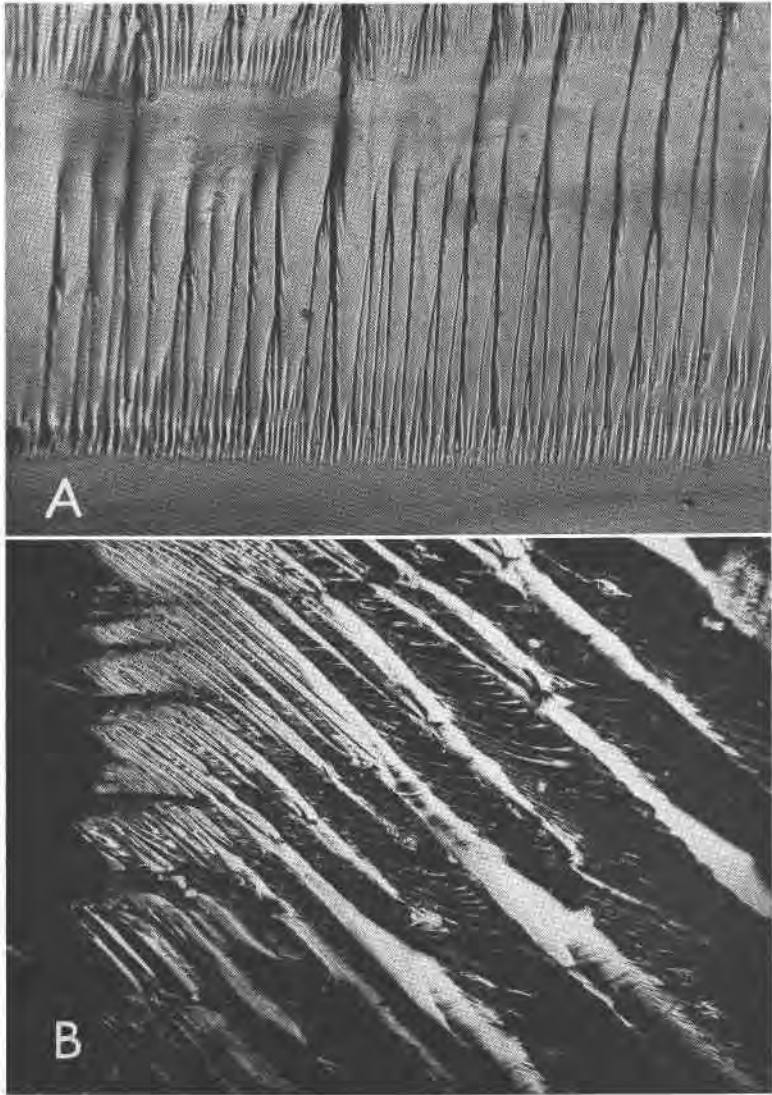


FIG. 3. Fractographs illustrating Type II patterns in synthetic ammonium dihydrogen phosphate crystals. (Above) Typical pattern caused by stress in a matrix exhibiting no directionalism. 100 \times . (Below) Type II pattern photographed with dark-field illumination and exhibiting fine-scale detail. 70 \times .

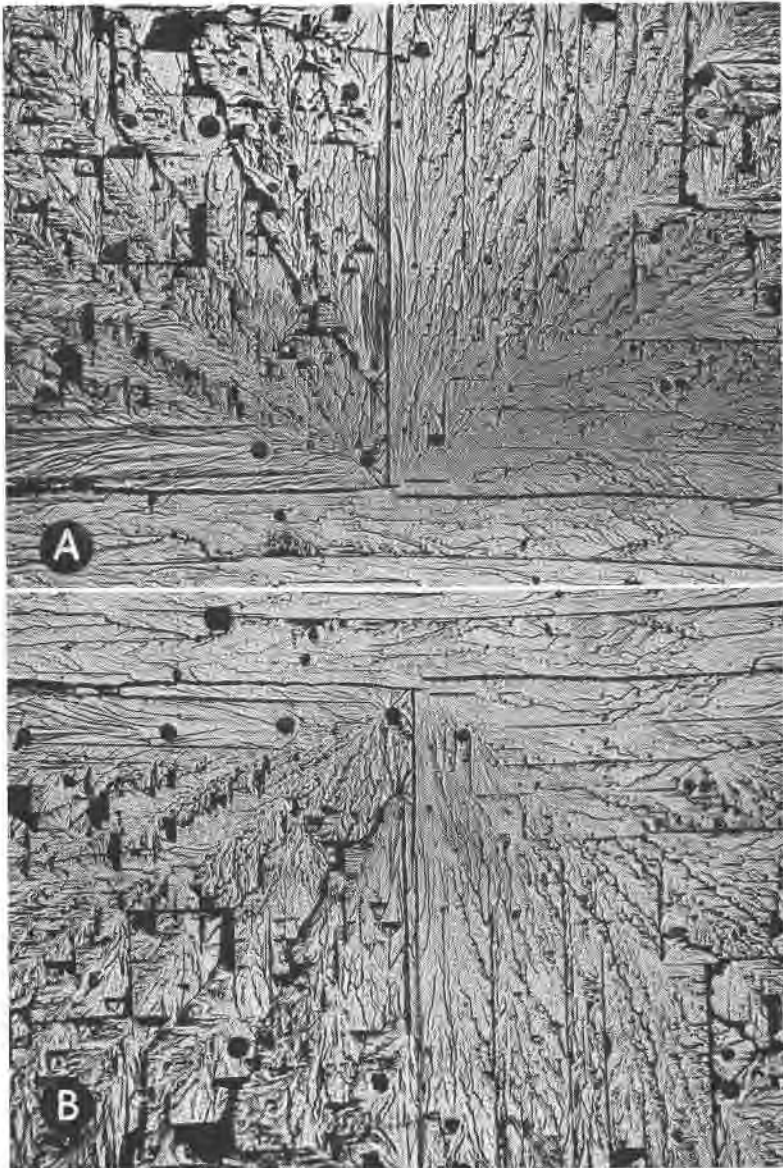


FIG. 4. Observe and reverse fractographs of a cleavage facet for a single grain within a polycrystalline specimen of isometric alpha iron made brittle by absorption of hydrogen gas. 235X.

face is (001), and the principal markings at 90° are intersecting (100) and (010) cleavages. Markings at other angles can usually be found on close observation to be stepwise composites of {100}, although some slip (gliding) activity on {110} or {112} is also possibly present. The subclass Type Ia identifies itself in the form of this crystallographic directionalism. Type Ib is strongly suggested in the minute blocklike formations, in conformity with the postulate of a mosaic or micellar structure (8), for cleavage would not suffer such perpendicular displacements if the path were not one of special weakness. Type IC can only be represented in this rather pure material, of course, by the pits and voids caused by inclusions of minute contaminating phases.

As for Type II, its patterns are virtually absent. The meandering or fanlike patterns could possibly be argued as a pattern of stress, but they can also be argued as an imperfection pattern referring to growth directionalism—the so-called “lineage” or dendrite pattern (16, 17). This distinction is often a subtle one; and the exact definition to be made between patterns from dendritic growth and those from stress remains to be determined. However, these are usually boundary cases of relatively minor importance. When they occur, experience with other systems where the origin of the pattern is quite certain will often allow a classification to be made. Where this cannot be done, the pattern will be so noted and left for subsequent study.

EXAMINATION OF MINERALS

In Table 1, the specimens used in this research are listed by name, crystal system, approximate composition, and source. In addition to these, others were also examined, such as olivine, talc, epsomite, thulite, magnesite, and quartz. Their description will not be included, principally for reasons of brevity, but also because of some photographic inferiority caused by such factors as poor reflectance and uninformative-ness of the patterns. The recorded work will suffice for the present purpose of introducing fractography as a possibly useful microscope technique and suggesting fields for subsequent special studies.

THE FELDSPARS

Plagioclase

Since one of the most important and most common of the mineral groups is that of the feldspars, plagioclase—the triclinic isomorphous series of Na-Al and Ca-Al silicates—is shown in Fig. 5. The specimen is labradorite; and the fractograph shows a system of parallel bands which can probably be accepted as albite twinning on the brachypinacoid {010}. The cleavage plane, which is the plane of the fractograph, can

TABLE 1. DESCRIPTION OF SPECIMENS

Material	Crystal System	Formula	Source*
1. Bismuth	Hexagonal-rhombohedral	Bi	Synthetic crystal (commercially pure)
2. Molybdenum	Isometric (BCC)	Mo	Climax Molybdenum Co. (vacuum cast)
3. Ammonium-dihydrogen phosphate	Tetragonal	$(\text{NH}_4)\text{H}_2\text{PO}_4$	Naval Research Lab. (synthetic crystal)
4. Iron (alpha ferrite)	Isometric (BCC)	Fe	Armco ingot iron
5. Plagioclase (Labradorite)	Triclinic	$(\text{Na}, \text{Ca})\text{Al} \left \begin{array}{l} \text{Si} \\ \text{Al} \end{array} \right \text{O}_8$	Labrador
6. Orthoclase	Monoclinic	KAlSi_3O_8	Carbon Co., Mont.
7. Muscovite	Monoclinic	$\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2$	Okanogan Co., Wash.
8. Fluorite	Isometric	CaF_2	—————
9. Fluorite	Isometric	CaF_2	Ferry Co., Wash.
10. Dolomite	Hexagonal-rhombohedral	$\text{CaMg}(\text{CO}_3)_2$	Stevens Co., Wash.
11. Limestone	—————	—————	Snohomish Co., Wash.
12. Calcite	Hexagonal-rhombohedral	CaCO_3	(twinned)
13. Calcite	Hexagonal-rhombohedral	CaCO_3	(optically pure)

* See acknowledgment. Further details on the specimens of minerals can be obtained from G. M. Valentine and N. W. Buerger.

probably similarly be accepted as a basal {001}. These planes were not specifically identified here; but a method has been developed in fractography which allows this to be done (18). That is, photographs of two intersecting cleavages are mounted on an appropriate model, and the families of the active planes are then directly determined from the positions of the traces in the three dimensions. This technique has already

resulted in a corrected determination for certain deformation phenomena in bismuth and antimony (18).

In Fig. 6 two further fractographs of plagioclase are shown. Several features of interest appear in the upper photograph. First, there is a pair of thin parallel lines running diagonally from upper left to lower right.



FIG. 5. Gridwork of albite twins in labradorite. 135 \times .

which can be tentatively judged as twins because of (*a*) their similarity to twin markings in the previous figure, and (*b*) the fact that the meandering "tear lines" are not noticeably influenced by their presence, as they would be if the markings were cleavages. Second, there are three further geometric markings forming a spearhead toward the left of the field, none of which is parallel to the previous set. Each of these three lines deflects the intersecting tear lines, suggesting a development prior to fracture; and one of these—the horizontal marking—is visibly a cleavage, which almost perfectly bisects the angle formed by the other two. As for these remaining two markings, they seem outlined by pits

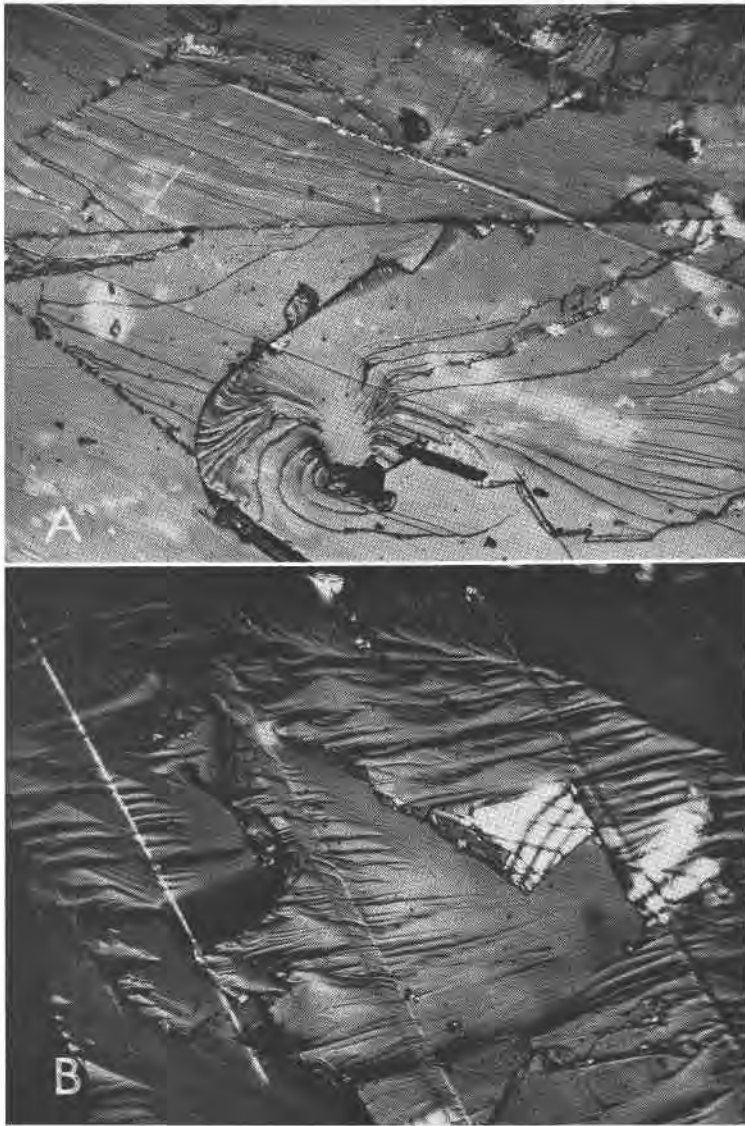


FIG. 6. Fractographs of labradorite. (Above) (a) albite twins, (b) intersecting cleavage, (c) parting on twin planes, (d) whorl pattern of tear lines nucleating at an inclusion, (e) fine-scale stepwise pattern in tear lines suggestive of mosaic structure and parting along minute twins, and (f) two intersecting pitmarked lines suggestive of a former growth face. 95 \times . (Below) Carefully shadowed surface detail showing (a) albite twins, and (b) fanlike patterns possibly relating to growth imperfection. 235 \times .

or inclusions, and may therefore represent remnant outlines of a growth face.

Thirdly, there are meandering markings—tear lines—which display a fine-scale stepwise structure suggestive of micellar imperfection. Fourthly, a large whorl appears in the lower center of the field, which appears to nucleate at the site of an inclusion or void. This whorl pattern may refer to growth imperfection, although it may also result from stress resolution around the pit (Type II pattern). It is an interesting feature that some of the tear lines completely reverse their direction. Before assigning this whorl to stress, reference should be made to similar patterns in ferrosilicon which definitely express imperfection from compositional changes in the neighborhood of the inclusion or void (19, 20).

Lastly, immediately to the right of this inclusion a short straight marking shows itself heavily shaded. It lies parallel to the bands ascribed to twinning and therefore may represent *parting* on the twinning plane. A similar instance will be shown later in calcite. Careful inspection of the stepwise meandering markings, just listed as the third feature, will show that these steps have their most prominent direction parallel with the bands, which suggests that a finescale weakness has developed from minute twins not otherwise observable. Such fine twins or “striae,” of course, are well known for plagioclase.

In the lower fractograph of Fig. 6, a field is shown for plagioclase which has an especially pronounced fanlike pattern combined with good examples of twinning. It is tempting to relate these fanlike markings to growth imperfection, but a reservation must be held for the effect of stress pattern.

In concluding the discussion of plagioclase, attention will be called to a special study which the preceding fractographs invite—an investigation of possible pattern changes across the constitutional range of this isomorphous plagioclase series. A marked sensitivity has been demonstrated in this respect for fractographs of metallic systems (19, 21).

Orthoclase

Passing now to the potassium aluminum silicate, orthoclase, the fractographic pattern in Fig. 7 can be tentatively accepted as typical.

No twinning is in clear evidence, and this seems to constitute the principal fractographic difference between the specimens of orthoclase and plagioclase examined in this research. The wavy bands of tear-line families, however, seem to stem from approximately parallel markings of unknown origin. These latter may relate to twinning; but in work with cast molybdenum metal (14), and in studies with bismuth-antimony

alloys, markings of this type have been related to pulsating solidification fronts during growth of the crystal. They may therefore similarly relate here to the growth history of the crystal. The tear-line patterns are elaborate in this material, and may also reflect growth imperfection, although this matter will require much further study.

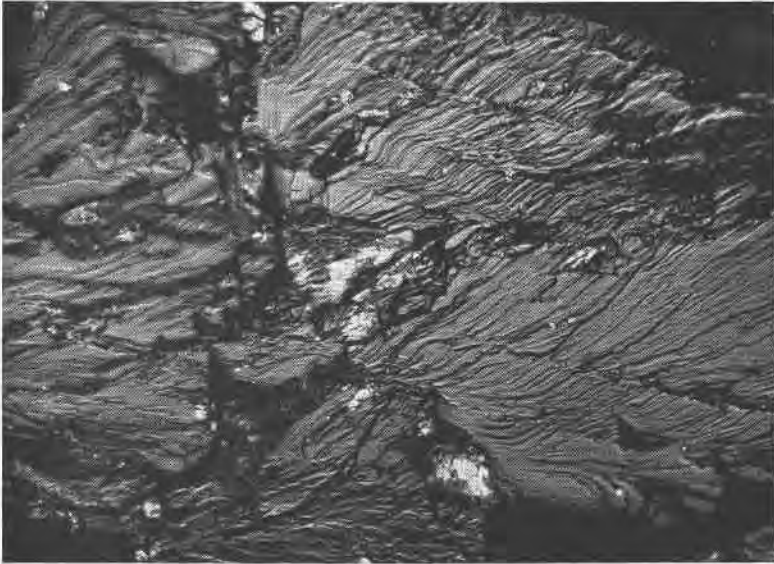


FIG. 7. Typical fractograph of orthoclase feldspar. 235 \times .

MUSCOVITE

Since the mica family provides a remarkable cleavage, a sample of muscovite was studied. The extremely perfect basal (001) cleavage renders the surfaces relatively free of intersecting markings; but Type Ic patterns may appear from interleaved phases.

In Fig. 8, two such patterns are shown. The upper fractograph discloses a number of very thin, transparent platelets trapped beneath the lamella of the cleavage facet. The focus is directed upon these, rather than upon the cleavage face itself. No analysis of these entrapped platelets was attempted; but their symmetry and transparency suggests that they are micaceous forms similar to the parent crystal.

In the lower fractograph of this figure, however, a beady phase appears which can readily be concluded to be foreign to the system. Again no analysis was attempted, the contribution resting with the fact that such phenomena are so clearly displayed.

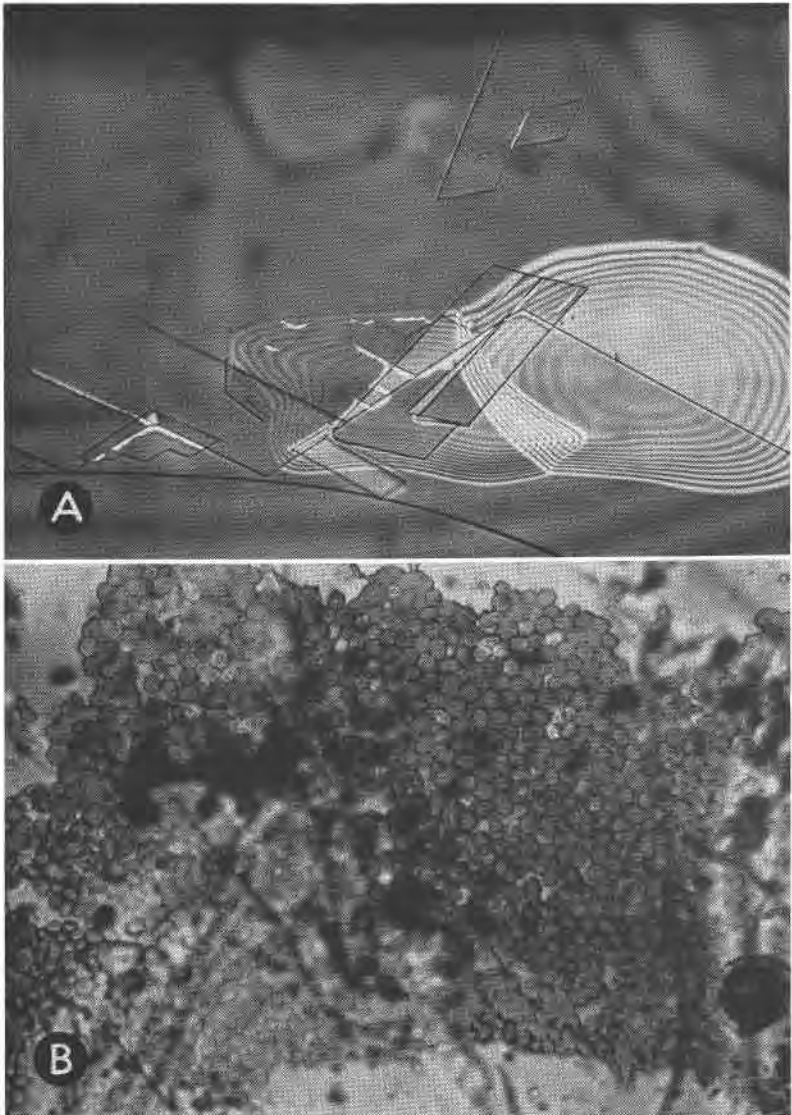


FIG. 8. Fractographs of muscovite. (Above) Interlamellar entrapment of minute crystals, probably of muscovite. 235 \times . (Below) Interlamellar entrapment of a foreign phase. 335 \times .

FLUORITE

Fluorite, CaF_2 , is an isometric mineral having such excellent cleavage on the octahedron $\{111\}$ and such transparency that the typical fractographic field is not particularly informative. Specimens from two different sources (Nos. 8 and 9 in Table 1) were studied; and a fractograph of each is presented in Fig. 9, principally for displaying some uncommon observations.

In the upper fractograph, one prominent intersecting cleavage appears, along with tear lines which are extremely regular in their disposition. This suggests that they conform to crystal weaknesses (Type I), rather than to stress resolutions (Type II). These weaknesses, of course, can be Type Ia crystallographic directionalism; but close observation of minute stepwise effects, both in a linear sense along tear lines and in a lamellar sense in the gradations of the cleavage levels, suggest a Type Ib mosaic effect.

In the lower fractograph, a most remarkable pattern appears. If this is attributed to growth imperfection of lineage or dendrite type, it is especially fascinating to find such an elaborate architecture within a matrix known to belong to a single crystal, hence having a common orientation; for it can only signify that the over-all molecular structure has maintained an approximately exact orientation, but that throughout the matrix there is distributed an imperfection pattern relating to crystal growth. The micellar theory accounts for this on the basis of a deposition of molecular clusters, or micelles, from the liquid state, such that the molecules within each micelle maintain the orientation common to the field, but a displacement is suffered among the micelles themselves. Fracture, strongly influenced by the path of maximum weakness, proceeds along the intermicellar boundaries and thereby depicts the imperfection architecture, of which this fractograph in Fig. 9 may be an excellent example.

THE CARBONATES

Once again a promising special study is suggested by the calcite group, whose various members clearly provide varying fractographic fractures. Here only a brief outline will be given.

Dolomite and Limestone

In Fig. 10, two fractographs are compared for dolomite and limestone, although the latter carries poor definition as a mineral. Nevertheless, both a relationship and a distinction are displayed. The dolomite shows the customary rhombohedral $\{10\bar{1}1\}$ perfect cleavage, with numerous

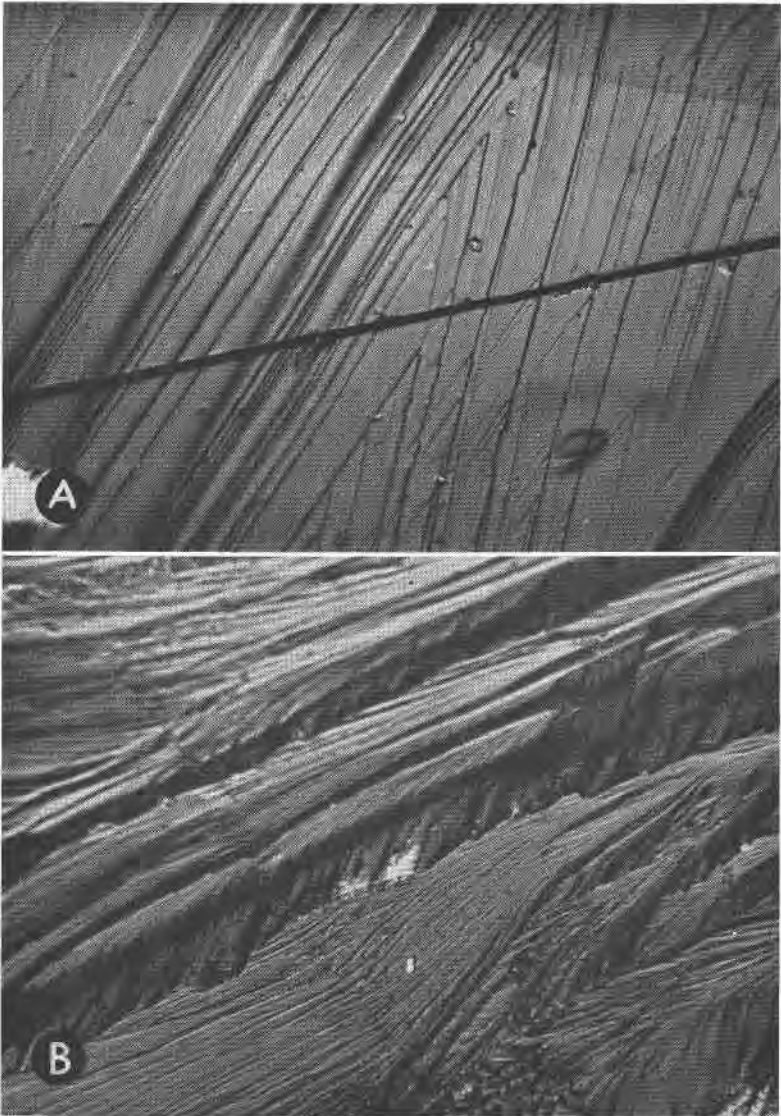


FIG. 9. Fractographs of fluorite. (Above) Intersecting cleavage and Type I tear lines. 235 \times . (Below) Phenomenal pattern believed to express growth imperfection. 315 \times .

bands or intersecting cleavages at angles closely approaching 90° . The limestone, on the other hand, shows only slight traces of these intersecting markings; and the cleavage is comparatively rough. This roughness shows itself as a mass of fan-like markings, or groups of tear lines; and



FIG. 10. Fractographs of dolomite (above) 335 \times , and limestone (below). 235 \times .

it is perhaps reasonable to conclude that these are structural patterns of growth imperfection, aggravated in this material by the greater complexity of its composition.

Calcite (Twinned)

Two samples of calcite were studied, one much twinned (No. 12 in

Table 1), and one optically pure (No. 13 in Table 1). Figure 11 presents a fractograph taken of the twinned sample No. 12 with the customary oblique illumination. Large tear lines or tear surfaces appear, overlain with an immense amount of fine detail. This detail somewhat divides itself into (1) a set of very delicate markings which are approximately parallel and vertical on the field, and which are strongly affected by the tear markings; and (2) a set of coarser markings which are also approximately parallel, running diagonally across the field and quite independent of the tear traverses. The angle between these two sets provides some similarity with the markings in the upper fractograph of fluorite in Fig.



FIG. 11. Fractograph of twinned calcite. 235 \times .

9. Still a third set can be found among the delicate traces, particularly in the upper right of the field; and these indicate a change of direction across the coarser markings consistent with the identification of the latter as twins.

In an attempt to decide whether the field contained a twin, a duplicate fractograph was taken using polarized light, though it will not be reproduced in the interest of conserving space. A light band then appeared running diagonally across the field from the base of the tear line on the right, but otherwise relating to nothing in the pattern. The diffuseness of this single illuminated band, however, suggested that it lay beneath the cleavage surface. Except for a uniform darkening over the whole portion of the distorted field, but little evidence appeared that any of

the pattern belongs to twinning. If twins are present, they must be fine superficial formations, formed at the instant of the formation of the new surface.

In Fig. 12, a fracture profile appears amid a pattern of tear lines and inclusions. Polarized light, in the lower fractograph, then reveals this profile to lie along a twin band; whereupon it can be presumed that the

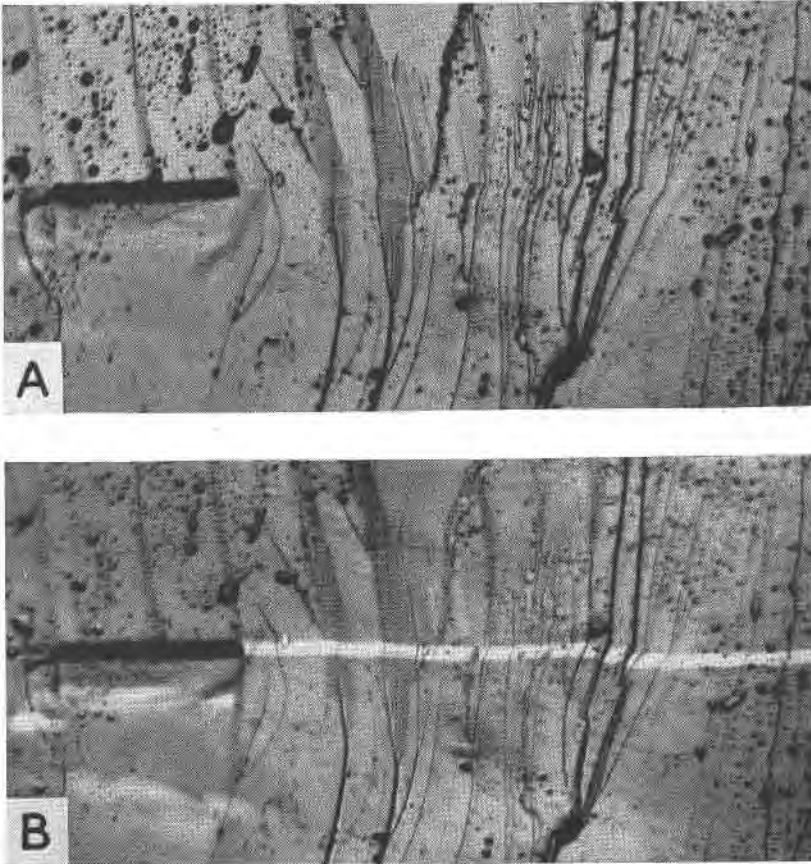


FIG. 12. Parting on a twinning plane in calcite. (Above) Oblique illumination. 350X.
(Below) Polarized light. 350X.

fracture represents parting on that twinning plane. Slight changes can be noted in the path of the tear lines as they cross this twin.

In Fig. 13, polarized light in the upper fractograph displays a prominent twin; and a rather remarkable displacement of this twin appears at the intersection with the heavy diagonal trace. Tear lines in the upper left of the field can be observed to deflect in crossing the twins; and very

close inspection of the fine striae in the lower right (perhaps difficult to observe in the reproduction) will show these similarly deflected. The lower fractograph in this figure displays a pattern which is quite remarkable, and not at all understood. Much of the detail in the central portion of

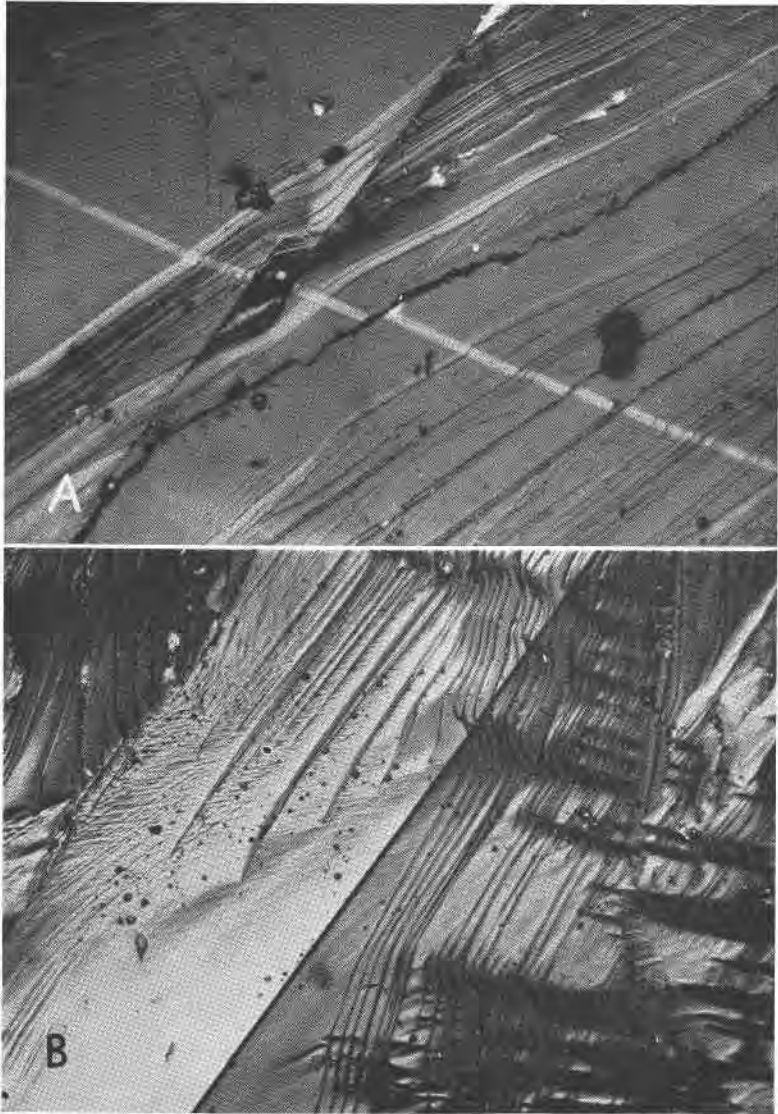


FIG. 13. Fractographs of twinned calcite. (Above) Polarized light. 235 \times . (Below) Oblique illumination of another field disclosing a phenomenal pattern. 235 \times .

the field, just to the left of the sharp diagonal line, is similar to the Type II markings illustrated in the previous Fig. 3; but the diagonal itself is puzzling, particularly because it simultaneously sections the field and yet imposes no alteration on the pattern at the upper right.

Four divisions can be noted in this fractograph, two to the left of the diagonal line, and two to the right. Counting these from left to right: (a) patterns 1 and 3 are similar, (b) pattern 4 shows strong evidences of twin structures, (c) none of the three boundaries dividing these fields

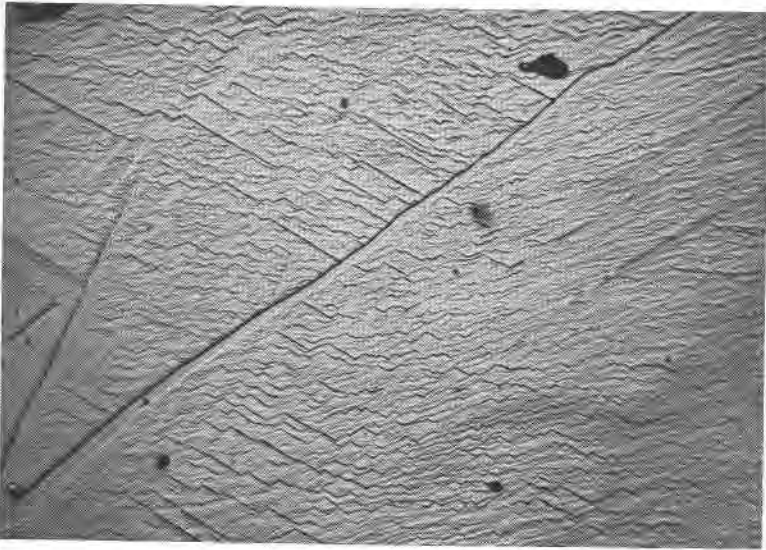


FIG. 14. Fractograph of optically pure calcite indicating lamellar imperfection. 235X.

is understood, and (d) fields 1, 3, and 4, particularly the latter, show strong indications of a lamellar imperfection structure, such as would derive from a micellar constitution.

Calcite (Optically pure)

By way of contrast, a laboratory sample of optically pure calcite (No. 13 in Table 1) was examined. The excellent cleavage provided visually flat fields, although at higher magnification considerable detail was exposed. The fractograph in Fig. 14 shows a field segmented by a diagonal boundary. The boundary shows a width, and the tear lines are short and markedly angular, disclosing the crystallographic directions to some extent. The surfacial lamellae outlined by these angular tear lines provide evidence for an inherent micellar imperfection structure even in this well developed crystal, and the matter should be explored

further from the standpoint of a possible relationship to optical and x -ray properties.

In Fig. 15, much evidence of fine-scale imperfection appears. The upper fractograph contains the same elaborate, delicate detail found in Fig. 11 for ordinary twinned calcite. This fine detail, then, can be due to twinning only if it develops during fracture. Strong indications of twinning stand in the chevron form of some of the markings; and the conclu-

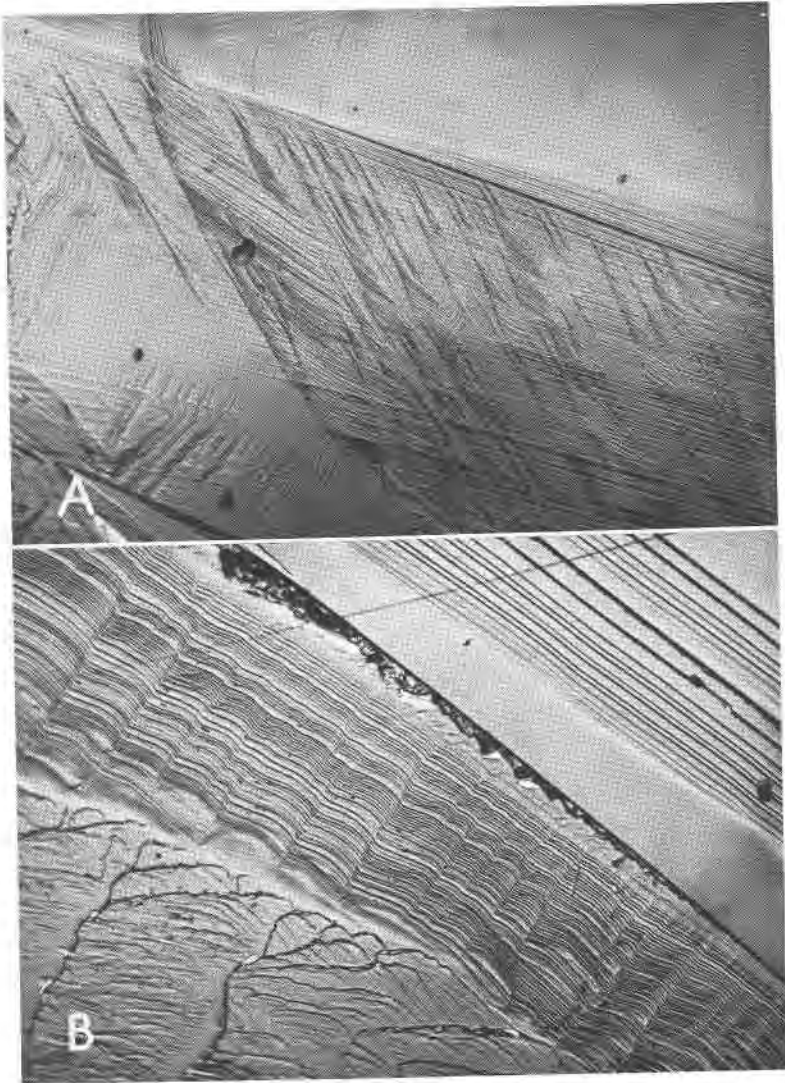


FIG. 15. Fractographs of optically pure calcite revealing fine-scale lamellae and suggesting superficial twinning similar to the calcite in Fig. 11. (Above) 100 \times . (Below) 235 \times .

sion can perhaps be drawn that much of this pattern involves twinning. Nevertheless, it will be recalled from the previous discussion that crossed nicols gave no strong indication of reoriented material. As suggested earlier, perhaps only a thin layer is twinned, a micelle or so deep. This would account both for the evidence of twinning and for the inefficacy of analysis by polarized light, also for the fine lamellar detail of the pattern.

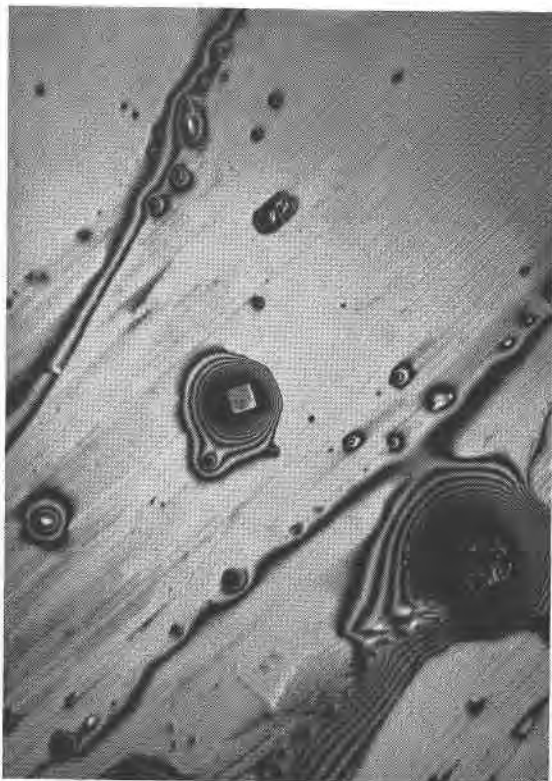


Fig. 16. Lamellar wall structure surrounding pits in optically pure calcite. 235X.

Attention is called to the particularly fine lamellar detail within the flat field at the top of the upper fractograph in Fig. 15. Several inclusions are also visible, two at left center apparently referring to a fluid phase.

In the lower fractograph of this same figure, a complex pattern is presented which reminds one of the previous Fig. 13 in the occurrence of several patterns within the one field. Most outstanding is the wavy formation in the center, which exhibits a lamellar form strongly suggesting micellar constitution. Note the columnar markings in this portion of the field approximately perpendicular to the heavy diagonal trace.

Markings in bismuth similar to these columnar ones are frequently observed, and their orientation similarly approximates a crystallographically significant position for that system (30° and 60°). Hence the present markings are not to be dismissed as Type II stress circumstances. It is also tempting to suggest that the tributary systems of tear lines in the lower left display the architecture of a cleavage cutting into the adjacent wave-like bank and following the indicated lamellar surfaces. This in turn raises the question why the field in the upper right, which is also visibly lamellar, exhibits a markedly different pattern. This cannot be answered by the present study; and the evidence will be left as material inviting further investigation. The thin straight trace across the top of the field should also be noted, for its surprising lack of deviation in crossing the several groups of markings shows the whole field of the fractograph to be quite flat.

As for the lamellar imperfection structure of calcite, even in the optically pure material, the concluding fractograph in Fig. 16 presents another aspect. Here pits appear, probably the seats of inclusions. The walls of these pits show a pronounced lamellar structure, hence one not influenced by deformation in this case, but by growth. The rings surrounding these inclusions are not to be confused with birefringence, such as occurs in the fractograph of muscovite in Fig. 8. The present rings are physical terraces which can be focussed upon.

CONCLUSION

From this preliminary study of several minerals and metals using the technique of fractography, the following conclusions can be tentatively drawn.

(1) Minerals, like metals, reveal a variety of detail upon their fracture facets which discloses structural features of the crystal.

(2) Fractographic patterns can be broadly classified as follows:

Type Ia: Patterns caused by crystallographic directionalism, whose traces therefore relate to the symmetry elements of the particular crystal;

Type Ib: Patterns caused by growth imperfection, or departure from the ideal directionalism of the crystal;

Type Ic: Patterns caused by extraneous or secondary phases;

Type II: Patterns caused by stress, resolving its waves and nodes in an effectively directionless matrix.

(3) All three subclasses of Type I disclose features of the crystal, and the patterns of both metals and minerals predominate in Type I.

(4) Type Ia discloses such crystallographic phenomena as (a) twin bands, (b) parting on twinning planes, (c) perfect or primary cleavage,

(*d*) intersecting primary and secondary cleavages, (*e*) unidentified striae perhaps representing fine-scale twinning, (*f*) markings possibly relating to historical growth faces, and (*g*) other markings.

(5) Type Ib refers specifically to crystal imperfection, which is an issue of much importance today. The fractographs of minerals, as of metals, provide abundant evidence for a mosaic or micellar constitution, particularly showing as fine-scale lamellae; and much evidence appears for lineage or dendrite-type growth imperfection during the aggregation of the micelles to form the crystal. This is true even of the more perfect crystals, such as optically pure calcite.

(6) Type Ic provides informative patterns for extraneous phases, specifically illustrated here for oxide and carbide in molybdenum, inclusions in numerous minerals, and two examples of entrapped interlamellar phases in muscovite.

(7) Type II only discloses features of the rupturing stress and is therefore more or less informationless so far as the crystal is concerned.

(8) Facets can be studied with any of the common types of reflected illumination; (*a*) vertical, (*b*) oblique, (*c*) dark-field, (*d*) sensitive tint, (*e*) polarized light, or (*f*) phase contrast. Oblique illumination is most useful, even for transparent minerals.

(9) Minerals can probably be readily distinguished from one another fractographically, as metals are, with further application of the technique; and some interesting special studies are suggested to determine, for example, whether members of an isomorphous series can be distinguished, since this has been accomplished for metals.

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APPENDIX

THE MICELLAR THEORY

One of the outstanding problems in researches on the solid state today is that of *imperfection structure*, and it is receiving first attention by laboratories in all parts of the world. Because fractographs directly disclose the internal structure of the crystal, a special significance attaches to the type of observations presented here. This Appendix is therefore added to clarify remarks in the text on *micellar structure*.

By "imperfection structure" is meant any phenomenon or disturbance which leads to a discontinuous atomic structure within an otherwise extended lattice. The fact of imperfection is no longer so much at stake, since most crystals are observably imperfect, but the inference from increasingly numerous observations is that crystals are by their very nature intrinsically imperfect. The history of the much involved argument on this subject can be found in Refs. 7, 8, 11, 12, and 16.

Briefly, it is now coming to be widely granted that crystals are minutely subdivided by discontinuities. Terminology for these discontinuities includes "dislocations," "Verhakungen," "Lockerstellen," "rifts," etc., whereas the structure itself is often referred to as "mosaic," signifying its subdivided nature.

In 1949, the senior author published a "micellar theory" which specifically accounts for the observed imperfection as the result of a cluster condition in the fluid state prior to solidification. If the theory is correct, a new field of research is opened for investigation of many important properties of solids not yet acceptably explained. Even if the theory is incorrect, the type of study promoted by its consideration is an important one.

TWELVE PRINCIPAL POINTS OF THE GENERAL THEORY

In brief topical outline of its principal aspects, the following twelve definitions summarize the general theory, from the particular standpoint of the solid inorganic state:

- I. That a universal condition of aggregation of atoms (or molecules) obtains within the liquid and/or gaseous states, probably at all temperatures, but certainly in a range just previous to solidification, this condition, because of its occurrence within an otherwise homogeneous constitution, being tentatively referred to as *isocolloidal*, and the aggregates as *micelles*.
- II. That the size of the individual micelle in the fluid state at any given temperature, other factors constant, approaches a preferred dimension expressing a balance among forces broadly represented as associative valence forces, dissociative thermal forces, and surface energy.
- III. That the shape of the individual micelle is a preferred crystal form for its system, having a high internal perfection modified externally by curvatures expressing the action of surface tensile forces, and perhaps further modified by adsorbed fractions of extrinsic phases in multicomponent systems.
- IV. That solidification has the nature of gelation, or agglomeration, of these micellar units—as opposed to the molecular concept

- of solidification—accordingly causing the solid state to inherit a virtually permanent and discrete substructure discontinuous on a scale of colloidal dimensions.
- V. That the approximate matching with regard to crystallographic orientation of the micelles during solidification provides the gross structure known as the lineage, dendrite, grain, or crystal.
- VI. That the mismatching of the micelles during solidification provides the imperfection structure of the crystal which has attracted so much attention and so little agreement in discussions on the solid state.
- VII. That this imperfection structure of the solid state therefore constitutes a universal and as yet unavoidable feature of all crystals larger than the unit micelle, such that the best precautions now known for crystal growth can effect no more than an improved matching of the micellar individuals.
- VIII. That imperfections in the solid state are accordingly of three general classifications:
- (a) *intermicellar*, which concern the fundamental fine-scale disjunctions between individual micellar faces.
 - (b) *interlineage*, which express the integrated intermicellar disjunctions occurring between two separately nucleated but commonly oriented aggregates or “lineages,” and
 - (c) *intergranular*, which relate to the surfaces of separately nucleated and separately oriented lineages or clusters of lineages (“grain” or “crystal”).
- IX. That the intramicellar forces are those primary molecular or valence forces which relate to the fundamental cohesive energy of the atoms in the perfect lattice structure, and upon which the so-called theoretical calculations of “ideal” crystal strength are based.
- X. That the intermicellar forces are principally secondary or adsorptive forces of markedly lesser magnitude, which determine the massive strength or cohesion of the “real” crystal and thereby explain the great discrepancy so characteristically noted between “real” and “ideal” crystals.
- XI. That the fundamental disjunction, the intermicellar adsorption face, upon which the entire imperfection structure of the solid state is thus based, involves a break in the regular lattice, not as vacant lattice sites predicated by most contemporary “dislocation” theories, but as a discrete and a highly persisting boundary for those micellar units from which it originates.
- XII. That these intermicellar boundaries provide (a) “slip planes”

for plastic deformation, whose minimum spacing is the unit micellar thickness, (*b*) cleavage surfaces, whose pattern is the fractograph, and (*c*) a universal presence of "internal surface" upon which all subsequent chemical and physical effects and constitutional changes must therefore be predicated.

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