

PYRITE OF UNUSUAL HABIT SIMULATING
TWINNING FROM THE GREEN RIVER
FORMATION OF WYOMING

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ABSTRACT

X-ray examination shows that pyrite crystals from the Green River Formation of Wyoming which have been referred to as twins are truly single crystals. The morphology of these crystals is strange, but they lack the features, prominence of the {210} form, or double sets of striations on cube faces which characterize the iron-cross twinning. The pyrite crystals are commonly coated with an exceedingly thin layer of sepiolite.

PRELIMINARY DESCRIPTION OF MATERIAL¹

Milton and Eugster (1959, p. 145) described the occurrence of pyrite in the Green River Formation in these words: "Pyrite is very abundant in Green River beds. Its crystal form is varied and unusual, much of it (in Utah) being in platy crystals of bizarre shapes, and in Wyoming, in extraordinary trillings [?] [plate 3b]." The photograph from which this plate was made is reproduced herewith as Figure 1.

Professor Milton has kindly provided about 75 small pyrite crystals comparable to those shown in Figure 1 for study. He states (letter dated Sept. 3, 1969) that the crystals are from the Westvaco mine near Green River, Sweetwater County, Wyoming, and that they "come embedded in trona, with shortite, etc., and are removed by simply dissolving the trona in water."

These pyrite crystals may correspond to the pyrite occurring with shortite in a drill core from the John Hay Jr. well No. 1, Sweetwater County, Wyoming, referred to by Fahey (1939). Later Fahey (1962, p. 27) described this occurrence of pyrite in these words:

An estimated 10 percent of the many hundreds of crystals of shortite examined contain either single cubes of pyrite as much as 2 mm across, or twinned crystals of pyrite of approximately the same size. Less commonly, crystals of shortite are seen with pyrite only partly embedded and protruding from one of the faces of a crystal of shortite. This would seem to indicate that the crystal of shortite grew around the older cube of pyrite.

Shortite was first found in a drill core from the John Hay Jr. well No. 1, not far from the Westvaco mine and penetrating the same section of the Green River Formation, the Wilkins Peak member, in which the mine operations are carried on. In the log of the drill core (Fahey, 1962, pp. 6-12) pyrite is recorded at 16 different levels, mostly with shortite.

¹ Material deposited in U. S. National Museum.

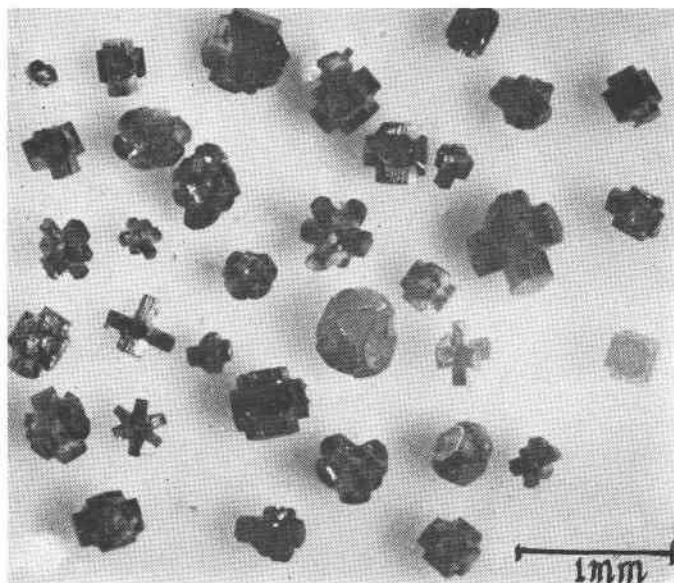


FIG. 1. "Pyrite crystals from trona mine. Wyoming"
(Plate 3*b* from Milton & Eugster, 1959)

All of the separated pyrite crystals supplied by Professor Milton have the appearance of being twins and one gets the impression that the material had been selected on this basis. Two other specimens from the same area, provided for study for other purposes, also contain pyrite. One of these is labelled "Leucosphenite with pyrite Wyoming." It contains a small sheaf of leucosphenite with the habit characteristic of this mineral as found at the Westvaco mine, attached to a rough, slightly mosaic, pyrite cube about 1 mm on edge. To this cube are attached many very small pyrite cubes *ca.* 0.1 mm on edge, plus a small loose pyrite cube and some bits, all somewhat tarnished. There is no indication of twinning in the habit. The other specimen containing pyrite is a semicylindrical piece of $2\frac{1}{2}$ -inch drill core 2 inches long, labelled "leucosphenite Wyoming." Pyrite is abundantly present, mostly as exceedingly small crystals embedded in the dark oil shale that makes up most of the core. It forms slightly larger crystals where it is present in veins of several minerals traversing the oil shale. The most conspicuous of these veins consist of loughlinite (identified by powder pattern) in asbestos-like fibers 5 mm or more in length. A bit of this adhering to a pyrite crystal became detached permitting closer inspection of the pyrite. It is a simple cube, about one mm on edge, with minor reentrants but lacking the striking features seen on the crystals in Figure 1.

The 75 pyrite crystals from the Westvaco mine showing considerable diversity of habit were inspected with a view to classifying them by habit types. Though some extremes are readily recognized there are gradations from one type to another. Certain features are common to all. The cube is always the dominant form. The principal cube faces are always bright and unstriated. All of the cubes have protrusions with bright faces at the ends but with the sides invariably striated. At one extreme the protrusions may be about as large as the central part of the crystal, but this is exceptional and only one or two such crystals appear in Figure 1. In these cases the whole edifice resembles an aggregate of seven cubes such as pictured by Chafranovski (1961a and b). The range of overall dimensions is not great, the average diameter of the larger crystals being of the order of 0.6 mm. The greatest dimensions of several measured crystals are from 0.31 to 0.57 mm.

TWINNING IN PYRITE

The only well established type of twinning in pyrite is the twinning by merohedry, ordinarily described as having the "twin axis [001] and the twin plane (011)," typically developed as "penetration" twins and sometimes called "iron cross" twinning. Only two orientations can be related by such twinning, no matter how many times repeated. It does not admit of the formation of trillings. Following Curien and Le Corre (1958), Curien and Donnay (1959), and Chafranovski (1961), the relations are conveniently described by the "black and white" symmetry symbol $4'/m\bar{3}2'/m'$, the primed operations relating the two parts of a twin.

Twinning on (111) was described by Nicol (1904), Goldschmidt and Nicol (1904) and Gaubert (1928), all of whom considered it exceptional. However, in his comprehensive summary of pyrite morphology Tokody (1931) refers to it as important. Both Tokody and Schaacke (1937) express skepticism as to the 46 other "twin laws" described by Smolar (1913), drawings for which were uncritically reproduced by Goldschmidt (1920).

The structure of the twin boundary or composition surface in iron-cross twins has been considered by Schaacke (1937) and by von Gliszczynski (1950). A preferable interpretation, avoiding the assumption of simple reflection at the twin boundary, is given by Strunz and Tennyson (1965) but none of these suggestions give any clue as to the morphological expression of the twinning.

DETERMINATION OF THE ABSENCE OF TWINNING

Donnay, Donnay, and Hurst (1955) have pointed out the advantages of precession goniometry in the study or identification of twins. They

deal particularly with what they call "neighboring twins," where two different twin laws, applied to crystal I, lead to nearly identical orientation of the twinned crystal II. The precession method is equally well adapted to the identification of twins, such as the pyrite iron-cross twin, in which there is only one orientation of the lattice, the same for crystal II as for crystal I.

In anticipation of finding iron-cross twinning in the material under study, ideal zero level precession patterns on the [100] axis of pyrite were prepared both for twinned and untwinned crystals. An untwinned pyrite cube from near Carson Hill, California (Pabst, 1931), similar in size to the Green River crystals, was used. A precession pattern corresponding to that of a twin was obtained from this crystal by means of a double exposure, recording a $(100)_0^*$ precession and a $(010)_0^*$ precession together, the crystal having been turned through 90° on [001] in the interval between exposures, corresponding to the operation 4' referred to above.

Initially four crystals representing the full range of habit variation were selected for examination by the precession method. From each only patterns matching exactly the simple $(100)_0^*$ precession pattern previously produced for reference from a single crystal were obtained. In addition one of the crystals was examined by precession patterns in two different [100] settings and another in four different such settings. The patterns obtained had the expected orientation and could be related to one another in the same manner as the striations can be matched on single crystal pyrite cubes (Steno, 1669). This fully establishes the fact that these are single crystals with only one orientation of the structure throughout.¹

PHYSICAL PROPERTIES OF THE PYRITE

Cell dimensions of the pyrite were determined from doubly quartz-calibrated $(001)_0^*$ Weissenberg patterns, using Cu radiation and a nickel foil covering the film. Such a pattern shows practically no background darkening due to "fluorescence." Patterns of this sort were run on a very small crystal and on a narrow sliver of it. Measurements, corrected for effective cassette diameter by means of the quartz calibration, were made on the well resolved α_1 and α_2 (1.54051 and 1.54433 Å) components of the high angle reflections. A Nelson and Riley extrapolation of the combined data leads to the cell constant 5.4175 Å with a standard

¹ Since the crystals can be precisely oriented on a reflecting goniometer equipped to take a standard X-ray goniometer head no X-ray adjustment of the crystals should be required. A single, one-hour, unscreened, 20° precession with unfiltered Mo radiation is more than adequate for making the distinction between a twinned and an untwinned crystal.

error of 0.0009 Å. This is just at the middle of the range found for pyrite of ideal composition (Sutherland, 1967). Microprobe analysis of several of the pyrite crystals by Dr. L. A. Taylor showed Ni and Co to be below the limits of detection, 0.1 weight percent, and the Fe:S ratio identical with that of synthetic FeS_2 ($a_{25^\circ} = 5.4175 \pm 0.0002$ Å, Taylor, 1970).

Since the individual crystals were too small for determination of density by Berman balance, it was necessary to resort to the method of the "basket for coarse powders." Determinations were made on lots of 5, 11, and 19 crystals which had been cleaned with warm dilute HCl, the largest lot having a weight of *ca.* 12 mg. These determinations led to the value 4.92 ± 0.05 , nearly two percent below the calculated value, 5.011+, corresponding to the cell constant previously determined. This unsatisfactory result is explained by microscopic observations on the crystals. Though broken crystals show that these pyrites are free of visible inclusions or voids and examination of polished surfaces by reflected light microscopy by Dr. Taylor showed no evidence of any phase other than pyrite, almost all of the crystals have remnants of a thin white or colorless coating that is impervious to attack by warm dilute HCl and that remains in the reentrant angles or even adheres to smooth surfaces.

IDENTIFICATION OF COATING ON PYRITE CRYSTALS

From some of the crystals, notably those with relatively large (111) faces (Figs. 3b and 3c), the coating could be removed in coherent sheets having a thickness of the order of 0.005 mm and a triangular outline corresponding to the outlines of the coated faces and slightly upturned at the edges. Several such sheets were detached and examined microscopically. They were found to consist of a finely felted mass of fibers with moderate birefringence and refractive index near 1.52. Rotation patterns from one such fragment yielded powder patterns without orientation effects fully indexed as sepiolite, in agreement with the optical observations.¹ Sepiolite has not been reported from the Green River Formation in Wyoming (Bradley & Eugster, 1969), though it forms thin beds near the top of the Formation in Utah (Bradley, 1929).

If a cube were one quarter covered by a coating with a thickness 1.50 of the cube edge and the densities of the cube and the covering were 5.00 and 2.23 respectively, the density of the combined material would be

¹ The particular fragment used for the X-ray pattern is a nearly equilateral triangle with upturned edges, 0.18 mm on edge and no thicker than 0.005 mm at most. Such a fragment would have a volume no more than 0.00007 mm³ and a weight, taking the density of sepiolite to be 2.23, of about 0.00016 mg. A six hour exposure in a 5.73 cm diameter camera with Cu radiation and Ni filter yielded a recognizable pattern. A more easily measured pattern was obtained by a twenty-hour exposure.

4.93. Thus it seems probable that the thin and discontinuous sepiolite coating may account for the low density values obtained for the pyrite.

MORPHOLOGY OF THE PSEUDOTWINS

Sixteen crystals, including six examined by X rays, were selected for detailed examination on a Stoe 2-circle reflecting goniometer. Two groups of five crystals each were examined by scanning electron microscope, an instrument which combines the advantages of high resolution and unlimited depth of focus with a great range of magnification. The second of these groups consisted of five measured crystals, including two examined by X rays. These five crystals had also been examined by binocular microscope and on a microscope with rotating stage while mounted on goniometer heads attached to a Goldfarb microgoniometer permitting rotation of a crystal on two axes while keeping it centered. In this way each crystal could be inspected from all sides at $100\times$ with illumination from any desired direction.

Thirty one scanning electron microscope photographs were taken at magnifications ranging from $24\times$ to $20,000\times$. One of these photographs, showing the second group of five crystals, is reproduced in Figure 2A. Details of the reentrants on each of these five crystals were photographed at $250\times$ or $640\times$ (Fig. 2b). An attempt was made at correlating the results of goniometric measurement and microscopic observations with features revealed by the scanning electron microscope.

On the basis of cursory examination of the entire lot of crystals supplemented by study of a limited number with the techniques outlined above it can be stated that all of the crystals have certain features in common:

1. The cube is always the dominant form.
2. The cubes always have reentrants along the edges. Alternatively one might say that there are rectangular protrusions over each cube face so that in the extreme the entire crystal corresponds approximately to a Chafranovski assemblage of seven cubes (except for physically dissimilar faces).
3. The sides of the protrusions are invariably striated and the striations are always normal to the end face on all four sides of the protrusion, as shown schematically in Figures 3a, b and c. The end faces of the protrusions are never striated. In a few cases they show minor mosaic structure of low plateaus with irregular edges. Some are marred by dirt or pitting, but the best faces are perfectly smooth. Such a face was seen to be a featureless plane even at $20,000\times$.
4. No indications of the $\{210\}$ form are found on any of the crystals not even in the continuum of reflections arising from the striations on

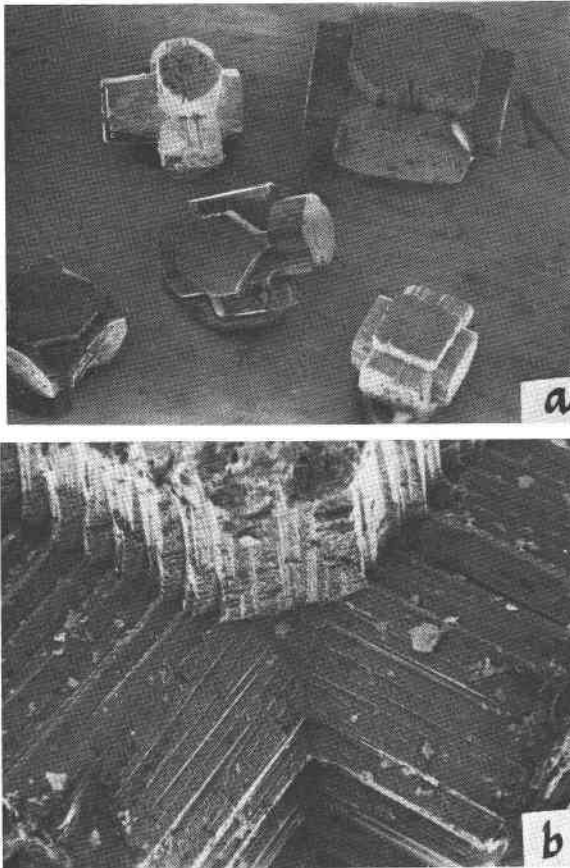


FIG. 2. a. (above) Scanning electron microscope photograph of five measured pyrite crystals from the Westvaco mine, Wyoming. Magnification 46X. b. (below) Scanning electron microscope photograph of corner of lower right crystal in upper picture. Magnification 640X.

some. This is in conformity with the directions of the striations on the edges of the protrusions. These do not correspond to the directions of striations commonly seen on pyrite cubes which are parallel to those cube edges at which the adjacent positive pyritohedron faces make an angle of $26^{\circ} 34'$.

Some features are observed only on certain types of crystals:

5. Octahedron faces are mostly found at the corners of the inner cube (Figs. 2b, 3b, and c) though in a few cases small octahedron faces were also seen at the corners of the striated protrusions. The more common octahedron faces are often coated with sepiolite even when most of the

crystal is free of coating. In several cases dull or coated octahedron faces are rimmed by bright reflecting borders corresponding in width to the depth of the reentrants (Fig. 3b).

6. Rarely reentrants do not extend along the full length of a cube edge but are terminated as shown at the front of the cube pictured in Figure 3b.

7. In many cases the striated surfaces yield no reflections other than those of $\{100\}$. In others these surfaces yield a continuum of reflections over a limited angular range giving rise to diffuse "spokes" extending in four directions at right angles from the $\{100\}$ signals seen through the telescope of the 2-circle goniometer. Within this continuum of reflection

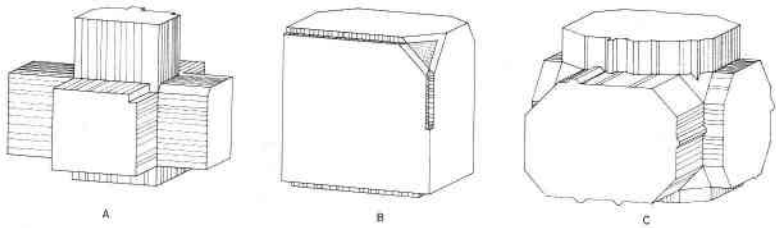


FIG. 3. Idealized drawings to emphasize certain variants of habit of pyrite crystals from the Westvaco mine, Wyoming. Each is based on observations on particular crystals and all are drawn to the same scale. The drawing at right represents a crystal with total width of about 0.4 mm. The striations in all cases are schematic only.

signals, but never from $[210]$, can sometimes be measured though particular faces are seldom directly recognizable. For example, in the case of the crystal pictured with only schematic representation of the striations in Figure 3A, the following reflections were recorded in the $[001]$ zone:

ϕ (meas.)	ϕ (calc.)	hkl
$1^\circ 49'$	$1^\circ 47\frac{1}{2}'$	1·32·0*
	1 51	1·31·0*
3 10	3 11	1·18·0*
4 50	4 46	1·12·0
6 41	6 20	190
7 12	7 $7\frac{1}{2}$	180

The forms marked by asterisk were not listed by Tokody (1931). It is not to be considered that they are here reported as new forms.

Among the five crystals shown in Figure 2a two show little or no continuum of reflections in the $[001]$ zone and three do show such continuum. There are no conspicuous features of the scanning electron

micrographs that can be correlated with this difference. Figure 2b shows the reentrant at a corner of a crystal which, on the 2-circle goniometer, yielded signals for the full complement of faces of $\{110\}$. Notes on the goniometric examination of this crystal read, in part: "Spokes visible in all $[001]$ zones, but become weak in regions *ca.* 30° from cube faces. No signals except from (110) faces in these zones." Notes on the microscopic examination at $100\times$ read: "No sepiolite crust seen. Some obvious dodecahedron steps associated with striations. The four end faces seen under microscope are all pitted or dirty but show no trace of striations."

8. In some cases the dodecahedron is developed in fairly regular fashion on all sides of the more conspicuous octahedron faces as seen on several crystals in the photograph, Figure 2a. Sometimes also recognizable strips of cube or dodecahedron faces determine offsets on the striated sides of the protrusions. Both these features are shown in Figure 3c which also shows a small (410) face at the right side of the front of the upper protrusion. The slight tapering of the protrusions on the crystal at the upper left in Figure 2a can be correlated with diffuse reflecting goniometer signals for unidentifiable trisoctahedra of high indices observed for this crystal only.

Goldschmidt (1920) reproduced 609 drawings of pyrite crystals plus Smolar's (1913) 60 questionable representations of twins. He did not indicate which drawings represent twins. But an inspection of the pictures shows that all of the twenty or so crystals that can be recognized as representing iron-cross twins, exhibit the $\{210\}$ form on each part of the twin, or are cubes having striations in two orthogonal directions on different sectors of the faces in a manner suggesting twinning. The absence of either of these features on the crystals described here may be considered morphological indication of the absence of twinning.

In minerals such as fluorite (111) twinning is commonly associated with a cubic habit giving the familiar penetration twins. If two or more cubic crystals grow in juxtaposition, such a twin may be simulated, and measurement is required to distinguish a true twin from an apparent twin. Among the 75 crystals inspected, there was one, *ca.* $\frac{1}{2}$ mm in dimensions, which on casual examination might have been taken to be a (111) twin. However, the direction of one of the faces of one cube, referred to the axes of the other cube, is only five degrees from the direction required by (111) twinning: a departure too small to be recognized with the unaided eye on so small a specimen.

Sunagawa (1957) and Sunagawa and Endo (1968) did not deal with twinning in their extensive studies of the variation in crystal habit of pyrite. Sunagawa was able to establish a correlation between habit and conditions of formation, but the conditions he considered did not include

an environment such as that represented by the Green River Formation. He classified habits on the basis of the three dominant forms, {100}, {111} and {210}. The Green River pyrites described here correspond to only two of the 19 "typical combinations" pictured by Sunagawa (1957, Fig. 1): the cube and the cube modified by the octahedron. In no case does the octahedron become the dominant form or even comparable in importance to the cube, and the pyritohedron is never observed. Neither Sunagawa nor Tokody make any reference to pyrite crystals with regular development of reentrants along the cube edges.

In a letter of 3 April, 1970, to Professor I. I. Chafranovsky Professor J. D. H. Donnay carefully described the curious morphology of the Green River pyrite and suggested, as one of two hypotheses, that it is due to skeletal growth. In his reply of 5 May, 1970, Professor Chafranovsky agreed that this is a reasonable explanation.

AN EXCEPTIONAL CRYSTAL

Aside from (111) reflections, only reflections from planes in the [100] zones were observed on most of the measured crystals. One crystal, though also having protrusions or reentrants similar to those of the other crystals, has additional features so distinctive as to merit discussion (Figs. 4a and b).

The absolute orientation of this crystal was determined by means of precession patterns¹ in a manner analogous to the oscillation methods used by Onorato (1931) and Paulitsch (1949, 1951) for the purpose of distinguishing positive and negative pyritohedra. Before the use of X-ray diffraction, distinctions between positive and negative forms on pyrite were dependent on observations of etching, striations of pyritohedron faces and/or on thermoelectricity (Curie, 1885). Most commonly, the distinction has not been made or has been based on assumption.

On one side of this crystal [010], $[\bar{3}40]$, and [904] zones are developed, each on a different protrusion and each with a great number of faces. Of the 27 forms measured only half a dozen are listed by Tokody (1931). In the [010] and $[\bar{3}40]$ zones a continuum of reflections is seen over a considerable range indicated by heavy lines along these loci in the stereographic projection. Within these ranges some distinct signals can be recorded. Most of these would correspond to faces with high indices. The previously unrecorded faces in these zones are not recorded here but are plotted on the projection; the agreement of calculated and observed

¹ From space-group requirements, (120) reflections must be absent, whereas (210) reflections are sufficiently strong, even though Fe makes no contribution to them, to appear upon only moderate exposure.

angles is good in some cases. For the [010] zone, the faces shown in the drawing belong to well-known forms and have the measured and calculated ρ angles $56^{\circ}37'$ and $56^{\circ}19'$ for (302); and $29^{\circ}54'$ and $21^{\circ}48'$ for (205) respectively.

The [904] zone is developed in quite a different manner. No continuum is seen, each face yielding a separate reflection though not all of equal quality. Only four faces in this zone are shown in the drawing and indices are indicated for only two of them. The full listing of the faces in this zone shows some interesting relations which are given in Table 1.

TABLE 1. ANGLE TABLE FOR [904] ZONE.

<i>hkl</i>	designation	meas.		calc.	
		ϕ	ρ	ϕ	ρ
$\bar{4}$ 6 9	negative diploid	$-32^{\circ} 41'$	$38^{\circ} 54'$	$-33^{\circ} 41'$	$38^{\circ} 42'$
$\bar{4}$ 7 9	negative diploid	$-29 41$	$41 30$	$-29 44$	$41 51$
$\bar{4}$ 8 9	negative diploid	$-26 45$	$44 1$	$-26 33$	$44 49$
$\bar{4}$ 9 9	trisoctahedron	$-23 3$	$48 19$	$-23 58$	$47 35$
$\bar{4}$ 15 9	positive diploid	$-14 59$	$60 18$	$-14 56$	$59 53$
$\bar{4}$ 18 9	positive diploid	$-12 42$	$64 35$	$-12 31$	$63 59$
$\bar{4}$ 21 9	positive diploid	$-11 23$	$67 5$	$-10 45$	$67 10$
$\bar{4}$ 24 9	positive diploid	$-10 18$	$69 31$	$-9 27$	$69 42$

In Figure 4a the regions in which the positive pyritohedra and diploids fall are shaded. In two of the strongly developed zones positive and negative forms are almost equally represented, whereas in the $[\bar{3}40]$ zone negative forms do not occur, this zone being so situated that only a very small segment of it lies within the negative sector.

POSSIBILITY OF SIMILAR CASES

In view of the difficulty of establishing the presence or absence of twinning in pyrite without the use of X-ray diffraction, it seems probable that there are other instances in which single crystals of pyrite have been referred to as twins. Smithson (1956), in a paper on pyrite in sedimentary rocks, referred to "twinning" of "interpenetrating cubes in the Millstone Grit of Anglesey" without giving supporting evidence. Ellis (1947) described pseudomorphs or partial pseudomorphs of pyrite of curious habit from Mississippian limestone in Missouri as twins but reported no substantiating observations. Efforts to obtain specimens of these materials for examination have been of no avail.

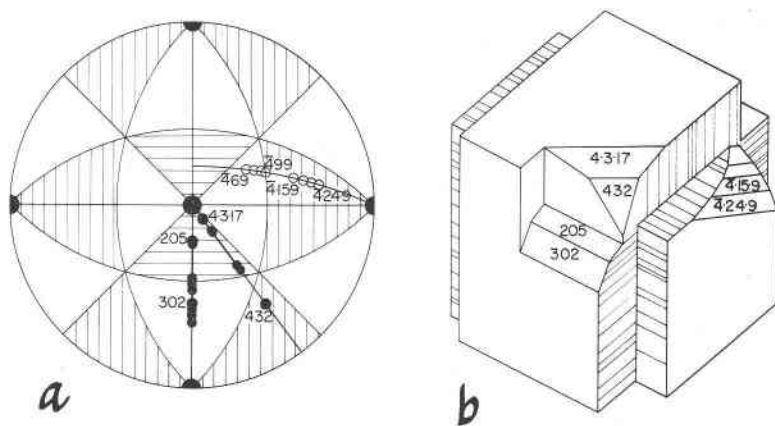


FIG. 4. A pyrite crystal of exceptional habit from the Westvaco mine, Wyoming. a. Stereographic projection with "positive" sectors shaded, the form $\{210\}$ being considered "positive." The orientation was established by precession pattern as described on p. 142. Corresponding axonometric drawing but showing only a few faces in each of the three developed zones, $\{010\}$, $\{340\}$ and $\{904\}$. The picture plane of this drawing has the coordinate angles ϕ $53^{\circ}8'$, ρ 49° . It is in the $\{340\}$ zone not far from (869).

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