CLEAVAGE IN PYRITE

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ABSTRACT

Cleavage in pyrite, analyzed by the method of Tertsch (1930) and by X-ray diffractometric studies of crushed samples, occurs parallel to {100} and, to a lesser extent, along {311}. Cleavage along {110} and {111} could not be verified. The X-ray method of studying cleavage here introduced—if compared to optical methods previously used on crushed quartz by von Engelhardt (1944). Bloss (1957) and Bloss and Gibbs (1963)—has the twin advantages of a great economy of time and of applicability to optically isotropic and opaque minerals.

INTRODUCTION

Palache *et al*, (1944, p. 284) attribute to pyrite a poor or indistinct {100} cleavage and an indistinct separation along {011} or {111} due to either cleavage or parting. The importance of this indistinct cleavage (Fig. 1) during the mechanical deformation of pyrite aggregates is emphasized by Ramdohr (1960), there being evidence that cataclastic structures develop in pyrite at stresses so low that they often produce no detectable changes in the accompanying minerals. Additionally the later localization of other minerals along these cleavage directions (Fig. 2), through oxidation, deposition or replacement, further heightens the interest in their nature and identity.

To a first approximation, the structure of pyrite resembles that of NaCl, differing in that (1) the bonding is prevalently covalent-to-metallic rather than ionic and, (2) the octahedra formed by the coordination of S about Fe have their four-fold axes not quite parallel to the crystallographic axes (*cf.* Fig. 16, Deer, Howie and Zussman, vol. 5, p. 129). To the extent that these differences are not important, the number of Na-Cl bonds broken per unit of area, if calculated for various crystal planes in halite, might indicate which of the comparable planes in pyrite represent the more likely planes for cleavage. For halite, the number of Na-Cl bonds broken per a unit of area equal to that of the unit-cell face—that is, a^2—is 4 along {100,} 5.37 along {210}, 5.66 along {110}, 6.03 along {311}, 6.53 along {211} and 6.93 along {111}.

EXPERIMENTAL STUDY OF PYRITE CLEAVAGE

Tertsch's method. The apparatus for quantitative studies of cleavage developed by Tertsch (1930, 1931, 1933, 1949) consists of a guillotine-like machine (Fig. 3) equipped with a

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razor blade or, interchangeable with this, an iron wedge with a 60° wedge-angle. For pyrite the wedge proved superior and was hence used in this study. The individual pyrite cube to be studied was then placed on the rotatable stage of the machine with the machine's guillotine blade poised over its center. After bringing the guillotine blade gently into contact with the cube's upper face—its lower face, of course, rests on the horizontal plane of the machine's stage—tiny lead pellets were gently added, bit by bit, to the pan suspended at the end of lever L in Figure 3. Ultimately enough compressional force was developed at the guillotine blade's knife-edge contact with the crystal's upper surface to cause the crystal to rupture along a surface approximately parallel to the plane of the guillotine blade. The
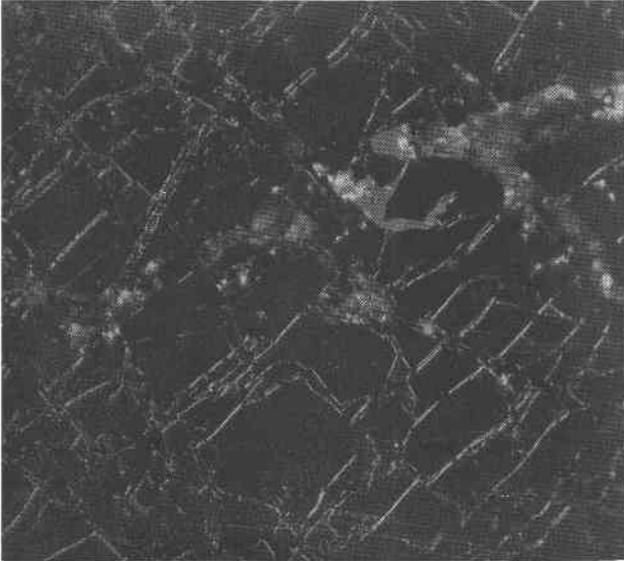

FIG. 1. Typical cubic cleavage in pyrite as shown in a polished section parallel to (100) for pyrite from Mine George near Horhausen, Siergerland, W. Germany. ($\times$165; dark field illumination).

magnitude of F, the force necessary to cause such rupture, could then be determined by multiplying (a) the weight in kilograms of lead pellets which produced rupture by (b) 5.133, the mechanical advantage imparted by the machine's lever system. Then (c) multiplication of the preceding product by 9.8×10^5 converts it to dynes, the cgs measure of force. Results in our study are reported with step (c) omitted. In other words the product of steps (a) and (b), a value in kilograms, is used as an index of the force required for rupture. Weight T in Figure 3 acted as a tare so that the weight of lever L and of its pan, if unloaded, exerted no force on the guillotine blade.

Using a binocular microscope, the more perfectly developed pyrite cubes were selected from a suite from Leadville, Colorado (University of Heidelberg's collection), and from a suite of unknown locality labelled S.I.U. 2103 in Southern Illinois University's collection. No attempt was made to improve the cube faces by grinding and polishing for fear of weakening the crystal in the process. Cube edges for the Leadville specimens ranged from 2 to 4 mm and for S.I.U. 2103, from 1 to 2.5 mm. Measured densities were 5.00_3 (Leadville
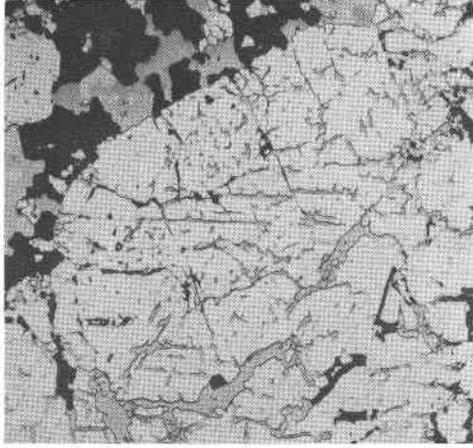
Fig. 2. Penetration of bournonite (gray) along the {100} cleavage directions in pyrite (white) as shown in a polished, random section for a Mine George specimen. (×165, reflected light).

specimens) and 5.00_6 (S.I.U. 2103). The surfaces resultant from rupture were seldom smooth, instead consisting of step-like surfaces parallel to {100} as in Figure 4. This complicated the measurement of A, the area for these ruptured surfaces, and, for simplicity, the cross-section of the cube as intersected by the plane of the guillotine blade was accepted as
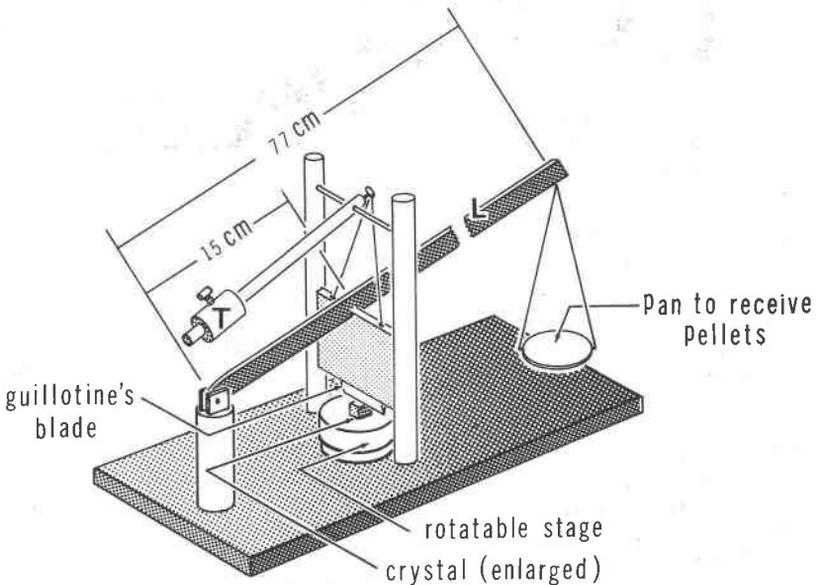

Fig. 3. Schematic diagram of Tersch's apparatus for measuring resistance to cleavage along particular directions in a crystal.
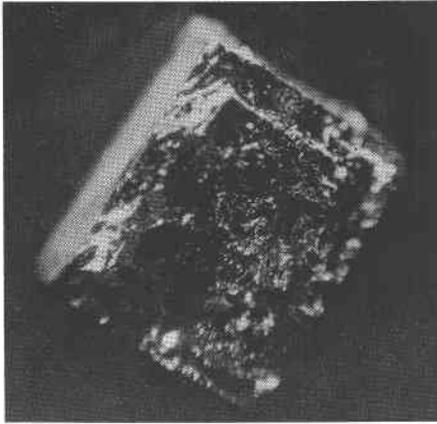
Fig. 4. Step-like cleavage parallel to {100} produced in a pyrite
cube from Leadville, Colorado.

this area. More force is required to rupture a large cube than a small one; hence the ratio F/A was chosen to represent R, a cube's resistance to rupture by the guillotine.

For 29 cubes from Leadville and 29 from S.I.U. sample 2103, each cube was oriented with a {100} plane parallel to the plane of the guillotine and its cleavage resistance R_0 was determined with the guillotine machine. Results (Fig. 5) reveal that cleavage resistance decreases as the area of the {100} rupture surface increases, such area being a direct function of cube size. Statistical analysis of the data determined the curve of best fit (dashed line in Fig. 5) to be

$$R_0 = 462 - 2373A - 14,292A^2 + 125,347A^3.$$

Eighteen of the points in Figure 5 represented cleavage resistance values which were considerably above the dashed line and the curve of best fit for these (solid line in Fig. 5) was determined to be

$$R_6 = 652 - 2746A - 23,816A^2 + 155,704A^3.$$

In the least-square calculations of the parameters for these curves, R was treated as the dependent variable and A as the independent one.

Within the size range investigated, the trend of the data and fitted curves (Fig. 5) indicate that the cleavage resistance, as here defined, decreases as crystal size increases. This perhaps expresses the increasing likelihood of crystals containing really extensive flaws as size increases. Such flaws may function like the "weakest link in a chain" to promote a premature breakage prior to attainment of the theoretical pressure value of ideal crystals. Thus, for very small crystals, the force necessary to produce rupture more likely approached the theoretical values. Actually, therefore, the pyrite cubes used here were not small enough, and better results would have been obtained using smaller crystals or even whiskers (see Stranski, 1942 and von Engelhardt and Haussühl, 1965).

For the S.I.U. 2103 suite, pyrite cubes less than 16 cubic millimeters in volume were again oriented with a face of the {100} form resting on the guillotine's stage but this time the cubes were rotated until a vertical face of the {100} form made an angle of 30° (or 45°) with the guillotine's blade, the resultant cleavage resistance values being now symbolized R_{30} (or R_{45}). For the 30° orientation, the guillotine blade is almost parallel to a {120} plane
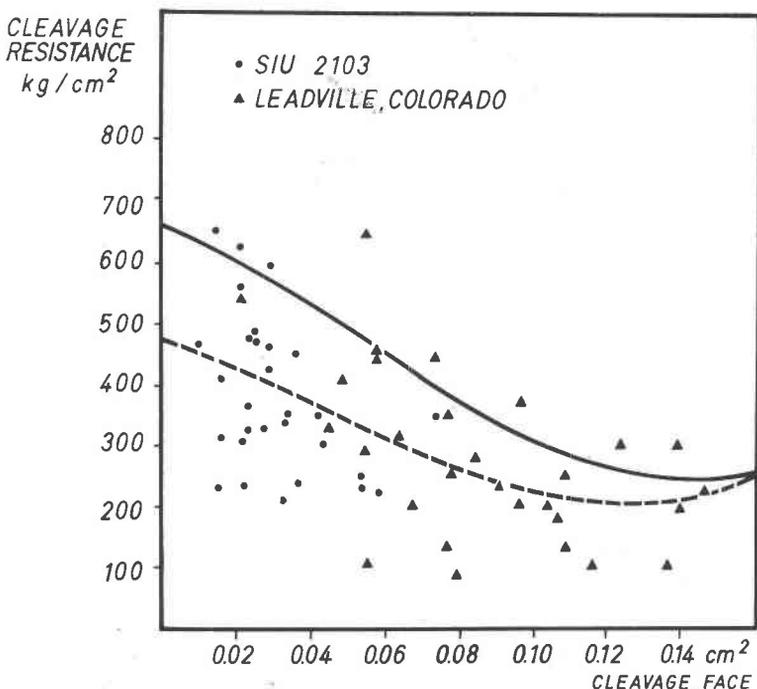
FIG. 5. Relationship between crystal size (expressed as area of cleavage face) and the cleavage resistance required by the Tertsch apparatus to produce rupture if the guillotine blade is parallel to a (100) face in pyrite. The triangles represent experimental data for cubes from Leadville, Colorado; the solid black circles represent data for pyrite labelled 2103 in the Southern Illinois University collection. The curve best fitting the combined data is represented by the dashed line; that fitting selected maximum values is represented by the solid line.

—a 26°34' orientation would have made it exact. For the 45° orientation, the blade is parallel to a {110} plane. Inspection of the surfaces of rupture disclosed no visible planar surfaces parallel to {120} or {110} but only irregular surface fractures with occasional steps parallel to {100}.

Diffractometer studies of crushed pyrite. Individual specimens of pyrite were crushed in a steel, Plattner mortar and one part of each sample was sieved onto a 27×46 mm glass slide. The latter was covered with a cardboard mask containing a square opening (2×2 cm) which confined the particles within a field standard for diffractometer studies. Ideally the particles (<44 microns in size) were regularly distributed over the 2×2 cm field with a density just short of that at which individual particles began to rest on each other. Hence, to the extent that cleavage directions exist in pyrite, preferred orientation is fostered in the sample because the particles would tend to rest with their flatter surfaces in contact with the glass slide. This preferred orientation would then cause the intensities of those peaks which correspond to cleavage planes to be larger, relative to other peaks, than for a sample in which preferred orientation is suppressed or eliminated. Interestingly, for slides prepared

as above to obtain maximum preferred orientation, the <44 micron-sized particles of pyrite adhered to the glass slide without need of a fixative.[1] As a precaution the glass slide and its adhering pyrite grains were exposed to ultraviolet radiation in order to eliminate the possibility of any static charges residual from the crushing or sieving.

Diffractograms of samples in which preferred orientation was minimized—that is, samples in which relatively few grains rest on cleavage surfaces—were now required for comparison with the results for maximum preferred orientation. Several methods for minimizing preferred orientation were attempted. In one, finely powdered pyrite, $\lesssim 5$ microns in size, was embedded in a vaseline-benzol mixture and packed into the standard aluminum holder to a thickness of 1.5 mm. Because pyrite's important peaks occur chiefly for 2θ values exceeding 35°, (iron radiation), the broad low-angle peak from vaseline did not seriously interfere. Judging from the relative equality in height of the 200, 210, and 211 peaks, the {100} cleavage had produced little preferred orientation in the particular mount of a pyrite from Rio Marina, Elba (Fig. 6A) made in the foregoing manner. Subsequently, however, mounts of this type were found to possess varying degrees of orientation which were introduced during the process of packing and levelling the vaseline-benzol-pyrite mixture into the sample holder by means of a spatula. Later studies showed that a more consistently effective method for reducing preferred orientation in diffractometer samples is that described by Flörke and Saalfeld (1955). In this case the pyrite particles are embedded in small plastic balls, the technique being briefly described in the accompanying paper by Bloss, Frenzel, and Robinson (1967). The Rio Marina pyrite, so treated, then yielded results as shown in Figure 6B.

In the diffractometer charts for pyrite shown in Figure 6, preferred orientation is low in *A* and *B*, high in *D* and *E*, and intermediate in *C*, this latter representing the chart for a normal mount—that is, one in which the finely ground pyrite is packed into an aluminum holder without adhesive or previous preparation. The increased prominence of the *200* peak as preferred orientation increases evidently results from the {100} cleavage. However, as preferred orientation increases and the *200* peak consequently increases in height, the *311* peak is not relatively attenuated to the same extent as the *111*, *210*, *211* and *220* peaks. Thus a lesser cleavage parallel to {311} is postulated whereas, based on this same evidence, cleavage parallel to {111} or {110} seems unlikely. The indistinct separation along {110} or {111} noted by Palache *et al* (1944, p. 284) is thus likely to represent a parting, if observed, rather than a cleavage.

The results summarized in Figure 6 were augmented through study of eight additional mounts of the Rio Marina pyrite, four representing Flörke-Saalfeld mounts, and four representing cleavage-controlled mounts like those from which Figure 6D was obtained. On each such mount, the Muller Micro 111 (Philips) unit at the Mineralogical Institute of the University of Heidelberg was used to obtain a count of the impulses per a fixed unit of time at the sites of each of the following six peaks: *111*, *200*, *210*, *211*, *220*, and *311*. Results served as an approximate measure of I_{hkl}, the intensity of the peak. For each mount the arithmetic average of the six peaks was determined and is here symbolized as I_A. The ratio of each individual peak to this average—that is, I_{hkl}/I_A—was next computed. Since different measurements from four mounts of each type were studied, four average values of I_{hkl}/I_A were available for each reflection and the mean of these four is entered in the columns headed I_{hkl}/I_A in Table 1.

Of the two types of mounts represented in Table 1, the Flörke-Saalfeld mounts showed greatest reproducibility. Individual I_{hkl}/I_A values for a single mount deviated within an average of 2.3 percent from the arithmetic mean of these values for all four mounts. Large

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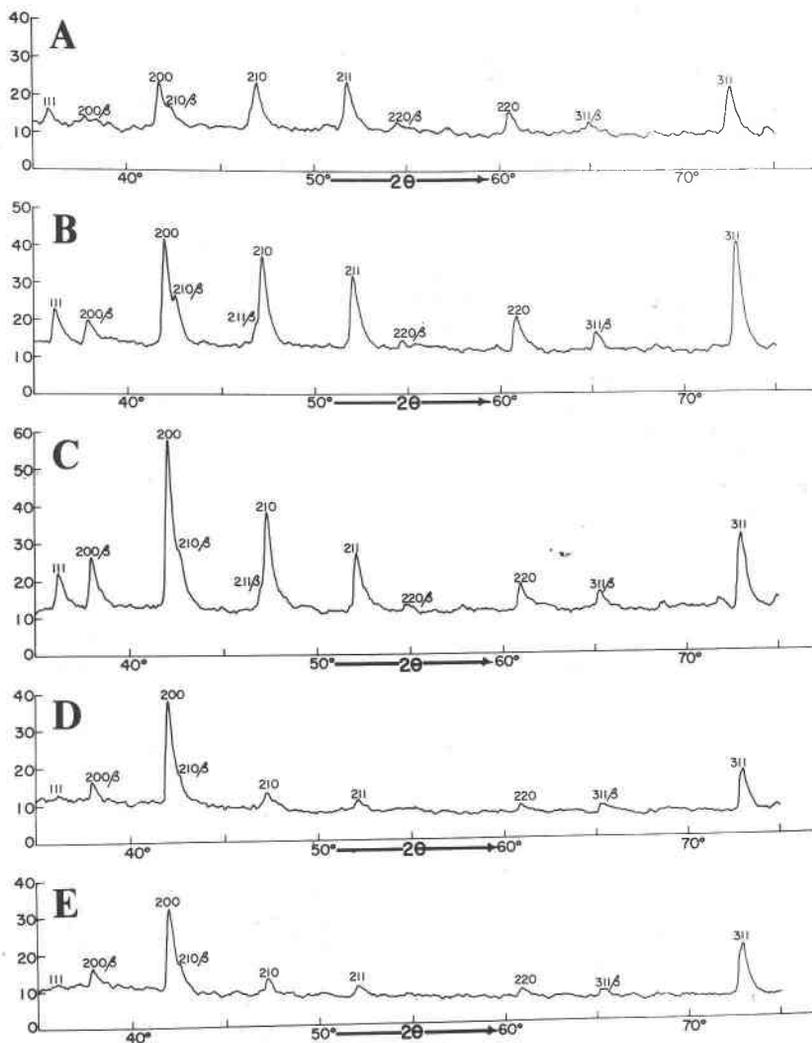[1] A NORELCO-type diffractometer with a horizontal axis was used.

Fɪɢ. 6 A–D. Reproductions of diffractometer charts for crushed pyrite from Rio Marina, Elba. (A) ≲5 micron sized, embedded in a vaseline-benzol mixture and packed in standard aluminum sample holder. (B) ≲5 micron size embedded in small plastic spheres as prepared using the Flörke-Saalfeld method. (C) ≲5 micron size particles packed in standard aluminum sample holder in the routine manner. (D) <44 micron particles sieved onto a glass slide so as to maximize preferred orientation. Note that the line joining the tops of peaks 200, 210 and 211 slopes increasingly downward to the right—from *B* to *C* to *D*—as preferred orientation increases. (E) Pyrite S.I.U. 2103 sieved onto glass slide to maximize preferred orientation.

TABLE 1. EFFECT OF PREFERRED ORIENTATION ON THE RATIOS I_{hkl}/I_A FOR PYRITE FROM RIO MARINA, ELBA. (Iron radiation)

	I_{111}/I_A	I_{200}/I_A	I_{210}/I_A	I_{211}/I_A	I_{220}/I_A	I_{311}/I_A

MINIMAL ORIENTATION: ~5 micron crystallites, Flörke-Saalfeld preparation (Measurements on 21 diffractometer charts from four sample preparations)

	I_{111}/I_A	I_{200}/I_A	I_{210}/I_A	I_{211}/I_A	I_{220}/I_A	I_{311}/I_A
Mean value	0.49	1.51	1.13	0.93	0.65	1.29
Mean deviation (%)	2.2	1.9	1.6	1.3	2.3	1.4

PREFERRED ORIENTATION: <44 micron crystallites sieved onto glass slide (Measurements on 15 charts from four sample preparations)

	I_{111}/I_A	I_{200}/I_A	I_{210}/I_A	I_{211}/I_A	I_{220}/I_A	I_{311}/I_A
Mean value	0.57	2.68	0.64	0.49	0.05	1.56
Mean deviation (%)	22.7	8.5	18.3	20.2	44.8	15.7

deviations existed between the I_{hkl}/I_A values obtained for a given peak on the four glass slides on which a strong cleavage-controlled preferred orientation existed. The large deviations of this latter type of mount reflect the sampling problem which arises because in the mounts it is necessary to use larger sized fragments (to 44 micron sizes) and distribute them sparsely on the slide (to avoid overlap).

Despite such sampling problems, the intensity measurements summarized in Table 1 appear to permit valid conclusions. For example, as preferred orientation was fostered, I_{200}/I_A increased from 1.51 to 2.68, a clear indication of {100} cleavage. In spite of this large increase in I_{200}/I_A, the value I_{311}/I_A rose from 1.29 to 1.56. Thus a cleavage along {311}, although a poor second to the {100} cleavage, probably exists. A {111} cleavage remains moot; the value I_{111}/I_A increases slightly but perhaps not significantly. On the other hand the values I_{210}/I_A, I_{211}/I_A and I_{220}/I_A decreased significantly as preferred orientation on the {100} and, less importantly, on the {311} faces occurred. Thus {210}, {211} and {110} are not likely as directions for cleavage in pyrite. Interestingly the value I_{220}/I_A decreased to the very small value, 0.05. This further supports the conclusion, already drawn from the Tertsch method of study, that {110} is not a cleavage direction even though the bond density calculations might lead one to expect it to be superior to {311}.

CONCLUSIONS

1. The {100} cleavage in pyrite is sufficiently pronounced to cause a degree of preferred orientation in the powder mounts normally used in X-ray diffractometers.

2. A poorer cleavage occurs along {311} but cleavage along {111} remains in doubt.

3. No cleavage exists parallel to {210}, {211} or 110]. The indistinct separation occasionally reported along {110} is likely to be a parting, perhaps localized along the {110} twin plane which sometimes occurs.

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