

STUDIES OF POLISHED SURFACES OF PYRITE, AND SOME IMPLICATIONS

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PART 1. THE OPTICAL ANISOTROPISM OF PYRITE²

ABSTRACT

In studying a large number of carefully polished surfaces of pyrite it has been found that this mineral is, in the general case, optically anisotropic. Interference colours are blue-green to orange-red, and bear a constant relationship to crystal morphology. On cube faces maximum green or red is developed when cube edges are inclined at approximately 45° to the planes of polarisation of the nicol prisms. Pentagonal dodecahedral faces show similar interference colours, as do all other planes except the octahedral, which are invariably isotropic.

Heat treatment experiments show that anisotropism is not irreversibly affected by temperatures up to 570° C. It apparently bears no relation to temperature of deposition, nor is it affected by compositional variations. It may, however, be obliterated during polishing if harsh methods are used, and in fact pyrite may be rendered apparently isotropic, or anisotropic, at will, simply by varying the polishing method.

The suggestion is made that the double refraction is an inherent characteristic of the pyrite crystal, and that it is due to the low structural, as distinct from dimensional, symmetry of the mineral. It is thought that the optical properties are essentially unrelated to the thermal and electrical features investigated by F. G. Smith and found by him to depend on lineage structures.

Introduction

The widespread ability of pyrite to cause double refraction of incident light was first recognized by the writer in 1955, when, in polishing a variety of Australian pyritic ores using a new polishing method (see Part 2 of this paper), it was noted that almost all material examined was distinctly anisotropic.

Although there are a number of references to this property in the literature, it had clearly been regarded as rather unusual and anomalous, and the sudden general appearance of the feature in the Australian ores seemed a little surprising. These initial observations have been followed up with a detailed examination of a large amount of material and it has been found that pyrite is almost invariably doubly refracting, whatever

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²Part two follows on page 103.

its form or environment of formation, and that the feature is stable over the temperature range of stability of the mineral.

Previous Work

Bannister (1932) in discussing the distinction of pyrite from marcasite in nodular growths stated that 'A closer examination of two sections of pyrite nodules . . . revealed a very weak anisotropy. These sections possessed a higher polish than the others and neighbouring blades showed very faint pink and blue polarisation colours . . .' He suggested that this was a strain effect. Stillwell (1934) noted occasional faintly anisotropic pyrite in zinc-lead ore from Rosebery, Tasmania, and following Bannister considered the feature to be anomalous and due to strain. He suggested that it was accentuated by the subjection of the pyrite to the heat and pressure of a bakelite press. Smith (1940, 1942), in observing variation in the physical properties of pyrite, noted that it was occasionally distinctly anisotropic, but he considered the feature to be generally nullified by heating. Schneiderhöhn & Ramdohr (1931) make the observation that pyrite may be anisotropic as a result of arsenic impurity and Uytenbogaart (1951 and personal communication) notes that pyrite is normally isotropic, but is occasionally slightly anisotropic. He suggests, from observations by himself and other workers on arsenical pyritic ores in Scandinavia that the anisotropism is probably due to internal tensions caused by arsenic or some FeS surplus. Recently McAndrew & Edwards (1954) have noted very weak anisotropism in pyrite from Rum Jungle, Australia, which they thought might be due to a high nickel content (suggested by the presence of associated nickel minerals). Their *x*-ray powder photographs showed, however, that the material was close to "pure" pyrite and contained less than 0.5% nickel.

Observations on Pyrite

Altogether some 350 polished surfaces of pyrite, in pyritic ores, pyrite-bearing rocks and as crystals have been examined. That a large number should be considered was obviously necessary for statistical reasons, and an effort has also been made to take material of as many different ages and environments of formation and from as widely separated localities as possible. Among the ores is material from Archeozoic to late Mesozoic age, from orebodies of accepted hypothermal to epithermal type, from Australia, the Southwest Pacific Islands and from North America. Pyrite of sedimentary and of orthomagmatic origin has also been examined. All specimens were either not mounted at all, or were mounted in a cold-setting plastic at room temperature, and polishing was carried out with diamond abrasive on stationary or slowly moving hand pads, as described in Part 2 of this paper. There was thus no

TABLE 1. PYRITE IN PYRITIC ORES

Type of Ore	Locality	Number of Specimens Examined	Host Rocks (pre-metamorphic lithology)	Accepted Origin	Age
1. lead-zinc-copper	Mt. Isa, Queensland	15	Highly folded Proterozoic black shales	Hydrothermal replacement	Houghtonian
2. lead-zinc	Broken Hill, N.S.W.	2	Highly folded Archaeozoic pelitic rocks	"	"
3. copper	Mt. Lyell, Tasmania	6	Tuffs and conglomerate	"	Lower Carboniferous
4. copper-gold	Cobar, N.S.W.	9	Folded Silurian-Devonian shales, sandstones, tuffs	"	Middle Devonian
5. lead-zinc-copper	Rosebery, Tasmania	12	Lower Palaeozoic Shales and pyroclastic rocks	"	Lower Carboniferous?
6. "	Captain's Flat, N.S.W.	30	Upper Ordovician—Lower Silurian black shales and tuffs	"	"
7. "	Peelwood, N.S.W.	18	"	"	Lower Palaeozoic
8. "	Wiseman's Creek, N.S.W.	36	"	"	"
9. copper	Burruga, N.S.W.	28	Upper Ordovician—Lower Silurian black shales and tuffs	Hydrothermal	Lower Palaeozoic
10. copper-gold	Mt. Morgan Queensland	8	Devonian (?) lavas and sediments	Hydrothermal replacement	Late Permian
11. complex poly-metallic vein deposit	Yerranderie, N.S.W.	9	Upper Devonian lavas, tuffs and shales	Hydrothermal cavity filling	Upper (?) Carboniferous
12. copper	Ballade Mine, Diahot,* New Caledonia	10	Palaeozoic pelitic sediments	Hydrothermal replacement	Palaeozoic (?)
13. gold	Fern Hill, Diahot, New Caledonia	15	"	"	"
14. copper-zinc-lead	Nemou Mine, Diahot, New Caledonia	10	"	"	"
15. copper-gold	Honfleur Mine, Plaine des Galacs, New Caledonia	5	Palaeozoic tuffs	"	"
16. copper	San Jorge Island British Solomon Islands Protectorate	4	Mesozoic (?) shales and lavas	"	Upper Mesozoic (?)

TABLE I *cont'd*

Type of Ore	Locality	Number of Specimens Examined	Host Rocks (pre-metamorphic lithology)	Accepted Origin	Age
17. copper-zinc	Amulet, Quebec	1	Keewatin-type lavas	Hydrothermal replacement	Late Precambrian (?)
18. copper-zinc	East Waite, Quebec	1	"	"	"
19. "	Normetal, Quebec	2	"	"	"
20. copper	Horne Mine, Quebec	2	"	"	"
21. gold	Hollinger, Ontario	2	Keewatin volcanic rocks and porphyries	"	Algoman
22. copper-zinc-gold	Flin Flon, Manitoba	3	Andesitic lavas and pyroclasts, quartz porphyry—Keewatin	"	Pre-Cambrian (?)
23. gold	Pamour Mine, Ontario	1	Temiskaming greywackes and other sediments	"	Algoman
24. nickel-copper	Levack Mine, Ontario	2	Sudbury affiliations	Hydrothermal replacement or igneous segregation	Pre-Cambrian
25. gold	Kerr Addison, Ontario	2	Temiskaming (?) sediments	Hydrothermal replacement	Algoman
26. gold	Teck-Hughes, Ont.	2	"	"	"
27. gold	Macassa, Ont.	2	"	"	"
28. lead-zinc	Sullivan, B.C.	2	Late Pre-Cambrian sediments	?	Late Pre-Cambrian
29. lead-zinc-copper	Bathurst, N.B.	51	Lower Palaeozoic pelitic and pyroclastic rocks	?	Palaeozoic
30. gold	Norseman, W.A.	2	Basic Lavas, pyroclastics and intercalated iron formation and shale	Hydrothermal	Pre-Cambrian

*All material from New Caledonia kindly contributed by the Bureau Minier de la France d'Outre Mer

elevation of temperature and an absolute minimum of distortion during polishing.

The range of material, excluding single crystals, is shown in Tables 1 and 2. In all ores almost all pyrite cubes, particles and aggregates were doubly refracting, and this was invariably the case with the sedimentary and magmatic material. Colours shown varied between blue-green and orange-red—the colours mentioned by both Bannister and Smith.

These pyrite particles and crystals of the ores and other rocks could, of course, only be seen in random section. However throughout the material the range of colours shown was quite constant, suggesting that

TABLE 2. SEDIMENTARY AND ORTHOMAGMATIC PYRITE

No.	Locality	Number of Specimens Examined	Nature of Material	Host Rocks	Origin	Age
31.	Portland, N.S.W.	2	Cubes	Fine marine siltstone	Sedimentary	Devonian
32.	Hohoro, New Guinea	3	„	Carbonaceous estuarine sediments	„	Miocene
33.	Pt. Philip Bay, Victoria	1	Radiating (colloform) concretion	Calcereous marine shales	„	Tertiary
34.	Sydney, N.S.W.	2	Cubes	Lacustrine siltstone	„	Lower Triassic
35.	Lardeau Map Area, S.E. British Columbia	1	Slightly deformed cubes	Marine calcareous argillite	„	Upper (?) Proterozoic
36.	Broken Hill, N.S.W.	2	Cubes	Metamorphosed calcareous pelitic rocks	„	Proterozoic
37.	Kakabeka Falls, Thunder Bay, Ontario	1	Radiating concretionary	Animiki black shales	„	Pre-Cambrian
38.	Bumbo Quarry, Kiama, N.S.W.	1	Isolated cube	Monchiquite dyke	Orthomagmatic	Tertiary (?)
39.	Bathurst, N.S.W.	3	Cubes and irregular grains	Andesite—basalt lavas	Orthomagmatic	Upper Ordovician
40.	Blayney, N.S.W.	2	Irregular grains	Monzonite and intruded andesite	Magmatic and contact metamorphic	Lower Carboniferous (?)
41.	Major's Creek, N.S.W.	2	„	Aplite dyke	Orthomagmatic	„
42.	Prospect, N.S.W.	4	Cubes and irregular grains	Alkaline dolerite—gabbro minor intrusion (laccolith ?)	„	Tertiary

the double refraction was produced by an ordered and consistent feature, very possibly bearing a constant relationship to the crystal structure. Little or no sign of undulose extinction was observed, indicating that the double refraction was not simply a local and variable strain effect.

In order to see whether the anisotropism did in fact bear a consistent relationship to crystal faces a number of well-formed single crystals were then examined; the nature of these and the localities from which they have been taken is shown in Table 3. The following observations, given in outline in the table, were made:

(1) Double refraction is invariably distinct, and possibly shows its

TABLE 3. PYRITE EXAMINED IN SINGLE CRYSTALS

No.	Locality	Forms developed	Properties between crossed nicols
WO-1745	Horne Mine, Quebec	Cube	Anisotropic, colours blue-green, orange-red when cube edges in 45° position.
43	Helen Mine, Ontario
31	Portland, N.S.W.
44	Captain's Flat Mine, N.S.W.
45	North Wiseman's Mine, Bathurst, N.S.W.
507	McIntyre, Ontario	Cube and Octahedron	Cube as above, octahedral faces completely isotropic
281	McIntyre, Ontario
D-31576	Prospect, N.S.W.
46	Bellinger, N.S.W.
D-28281	Valla Mine, Urunga, N.S.W.	Octahedron	Isotropic
D-4159	Milhau, Aveyron, France
D-37431	Trevenna Mine, Nundle, N.S.W.
D-34451	Lobb's Hole, Yarrangobilly, N.S.W.	Cube and pentagonal dodecahedron	Cube as above, pyritohedra also doubly refracting
D-34452	Monroe's Mine, Zeehan, Tasmania
D-34453			
D-23679	Mt. Galena, Emmaville, N.S.W.	Pentagonal dodecahedron	All faces doubly refracting
D-20102	Franklin, New Jersey
WO-1786	Butte, Montana

*Prefix WO denotes collection of Department of Geology, University of Western Ontario; D the collection of the Australian Museum, Sydney; No. 507 and 281 are from F. G. Smith's material at Miller Hall, Queen's University.

greatest intensity, on polished cube faces and surfaces sawn parallel to these. With nicols nearly crossed (or, when double refraction is particularly pronounced, with the nicols completely crossed) the colours are blue-green and orange-red, the maximum or purest red or green being developed when the cube edges are at approximately 45° to the vibration directions of the nicol prisms. These colours, and the directional feature, have been found in every case for sections parallel to the cube face.

(2) Sections parallel to the octahedral faces are isotropic. Again both lightly polished natural faces and faces produced by grinding or sawing

parallel to 111 were examined. Where complete octahedrons were available, four non-opposing faces were polished to see whether some were isotropic, others anisotropic. In most cases all appeared completely isotropic. In one or two individual faces extinction was not complete, but double refraction was so slight it seemed most likely to be due to the production, during polishing, of a plane just off the 111, although a great deal of care had been taken to keep the polished planes parallel to the original faces.

(3) Pentagonal dodecahedral (pyritohedral) faces and sections parallel to them always showed distinct double refraction, with interference colours essentially similar to those shown by planes parallel to cube faces.

(4) No further different forms were available to the writer for examination, but random and oriented cuts were made in several groups of well-developed intergrowths, and simultaneous observations on the differently oriented crystals made. In every case the individual crystals showed up by contrasting colours between crossed nicols.

These observations are for the most part quite in agreement with those of Bannister and Smith. The former had noted faint pink and blue polarisation colours, and the latter had, in a cubic crystal from Leadville, Colorado, noted the features of cube and octahedral planes outlined in (1) and (2) above. These features are now, however, recorded as general.

Stability of the Anisotropism

(1) *The effect of heat.* The only person to have considered the possible instability of the feature is Smith (1940, 1942) who in his very careful and detailed investigation of the various physical properties of pyrite came to the conclusion that double refraction is shown by pyrite only until it has been subjected to temperatures in the vicinity of 135°C. Heat treatment above this point, he states, nullifies the feature, the transition being irreversible over short periods.

That heat treatment should have this effect was suggested to Smith by the changed appearance of pyrite after mounting in bakelite, and repolishing. He noted (1940) that some fragments polished by hand on cloth laps showed distinct anisotropism, but that these sections, after mounting in bakelite and mechanical polishing, were all isotropic. The maximum temperature of the bakelite moulding die was about 135°C and therefore, according to Smith, heating to this point was sufficient to destroy anisotropism. Later, in attempting to determine the exact temperature at which the change took place, Smith (1942) heat-treated a doubly refracting cube in an oil bath to temperatures in the 100°–214°C range, but with no apparent effect. The possibility that larger crystal

size might have had a dampening effect on the change was then tested (1942, p. 12) and the conclusion reached that 'the smaller the size of the crystal fragment, the more readily the transition takes place'—an erroneous conclusion in which, as will be shown later, polishing deformation was interpreted as a heat effect. Strangely Smith then cast aside his evidence of persistence of the feature above 135°C and came to the conclusion that a temperature approximating to this was the critical one.

To test Smith's assertion the writer has carried out the series of

TABLE 4. PYRITIC MATERIAL HEATED IN NITROGEN STREAM AT ATMOSPHERIC PRESSURE

No.	Locality	Nature of Material	Heating Time (room temp. 24° C.)	Maximum Temperature (2 hours)	Cooling Time (approx.)
31	Portland, N.S.W.	Isolated cube	4.75 hrs.	58° C.	1 hr.
31	"	"	4.25 hrs.	135° C.	1.5 hrs.
31	"	"	4.75 hrs.	200° C.	2.0 hrs.
31	"	"	4.75 hrs.	325° C.	3.0 hrs.
31	"	"	4.0 hrs.	415° C.	3.0 hrs.
31	"	"	4.0 hrs.	550° C.	3.0 hrs.
47	Mt. Morgan, Queensland	Irregular large grain from copper-gold ore	4.0 hrs.	415° C.	3.0 hrs.
D-34452	Munroe's Mine, Zeehan, Tas.	Well-formed pyritohedron	4.0 hrs.	570° C.	3.0 hrs.
48	Mt. Isa, Queensland	Well-formed cubes with galena pressure shadows in dark shale	4.0 hrs.	572° C.	3.0 hrs.
49	Wiseman's Creek,	Pyritic Cu-Zn-Pb ore	2.0 hrs.	500° C.	2.0 hrs.
50	Captain's Flat, N.S.W.	Pyritic Cu-Zn-Pb ore	1.25 hrs.	515° C.	2.0 hrs.
51	Hercules Mine, Rosebery, Tasmania	Pyritic copper ore	2.5 hrs.	500° C.	2.0 hrs.
52	Franklin, New Jersey	Pyritohedron from Australian Museum Collection	1.5 hrs.	500° C.	3.0 hrs.
53	Valla Mine	Octohedron	1.5 hrs.	500° C.	3.0 hrs.
54	Yerranderie, N.S.W.	Coarse pyritic silver- lead ore	1.5 hrs.	505° C.	3.0 hrs.
55	Cobar, N.S.W.	Pyritic copper ore in black shale	1.0 hrs.	500° C.	2.0 hrs.
56	Climax, Colorado	Pyritohedrons in rhodocrosite	5.5 hrs.	530° C.	3.0 hrs.

heating experiments summarized in Table 4. Unmounted material, previously determined as anisotropic, was placed in a glass container through which a stream of nitrogen could be passed continuously. The whole was placed in a furnace in which temperatures could be controlled to about plus or minus 5°C.

As shown in Table 4, the pyrite was raised to a variety of temperatures, and both heating and cooling times were varied. In some cases the material was quite coarse (single crystals up to 2.0 cm. across) and in others fine. Where temperatures above about 500°C were attained it was impossible to prevent some slight oxide tarnishing, and specimens D34452 and 56 broke into several fragments from which, however, some large enough for repolishing could always be selected. In no case was any of the material mounted at any time.

When lightly "touched up" on a stationary diamond pad after heating, all pyrite was still anisotropic—to a degree which could not be distinguished from that observed prior to heating. It was thus clear that for some cases at least heat treatment had no major irreversible effect on the crystal structure.

TABLE 5. PYRITE HEATED IN VACUO TO 500°C.

No.	Locality	Type of Material	Origin
WO-1745	Horne Mine, Noranda, Quebec	Group of intergrown cubes	Hydrothermal
WO-617	Flin Flon Mine, Manitoba	Massive, very fine grained pyrite	„
WO-1786	Butte, Nevada	Intergrown well crystallised pyritohedrons	„
57	Helen Mine, Ontario	Intergrown cubes	Altered Hydrothermal ?
58	Thunder Bay Dist., Ontario	Spherical concretion	Sedimentary—black shales environment
59	No locality	Two large cubes, intergrown	Unknown

To test the idea further, but in a more carefully controlled way, six North American pyrites, five from well-known localities, one a well formed intergrowth of two cubes from an unknown locality (see Table 5), were polished unmounted and found to be doubly refracting. X-ray powder photographs all showed a normal pyrite pattern. Each specimen was then separately sealed in an evacuated pyrex tube, placed in a furnace and raised from room temperature to 500°C over four hours. This temperature was held for 20 hours, and then cooling back to room temperature allowed over about 3 hours. Heat-treatment again pro-

duced no detectable change in the double refraction, and further powder photographs of each indicated no change in crystal structure.

Examination of "High" and "Low" Temperature Pyrite

As Smith had considered that the optical properties of pyrite might serve as a further geothermometer (1942, p. 17) it was suggested to the writer by Professor J. E. Hawley that some pyrite from the McIntyre Mine, Porcupine District, Ontario, the temperature of deposition of which had been determined by Smith on the basis of his electrical conductivity methods, might be examined. Twenty-four specimens, listed

TABLE 6. PYRITE FOR WHICH TEMPERATURE OF DEPOSITION HAS BEEN DETERMINED BY F. G. SMITH. INDEX NUMBERS AND ALL OTHER DATA ACCORDING TO SMITH

Sample Number	Mine Level	Distance from porphyry-sediment boundary	Vein Number	Temperature of deposition as determined by Smith	Description of material
491	5225	500' inside porphyry	10	610° C.	Irregular crystals in quartz, pyrrhotite and chalcopyrite
493	5225	450' inside porphyry	10	575° C.	Irregular masses in porphyry
317	2125	350' inside porphyry	10	620° C.	Irregular areas in quartz vein
461	4925	300' inside porphyry	13	500° C.	Massive veinlet in quartz-feldspar vein
427	4775	270' inside porphyry	10	475° C.	Disseminated in grey schist
458	5375	220' inside porphyry	25	125° C.	Irregular cubes in quartz vein
462	2375	70' inside porphyry	10	510° C.	Cubes in grey schist
507	4925	30' inside porphyry	21	450° C.	Scattered cubes in grey schist
164	3750	Contact	15	410° C.	Small cubes in quartz
413	4625	5' outside porphyry	25	510° C.	Thin veinlet in black slate
408	4625	10' outside porphyry	25	430° C.	Irregular groups in black slate
145	1500	50' outside porphyry	5	630° C.	Irregular particles in quartz
320	1750	50' outside porphyry	7	160° C.	Striated cubes along border of quartz vein
251	200	60' outside porphyry	13	125° C.	Large cubic crystals in quartz
480	3875	70' outside porphyry	21	125° C.	Massive veinlet in greenstone
154	500	100' outside porphyry	5	175° C.	Minute cubes in quartz stringer
395	3250	110' outside porphyry	14	150° C.	Small cubes in grey schist
484	3500	130' outside porphyry	13	125° C.	Disseminated cubes in greenstone
178	1625	220' outside porphyry	10	125° C.	Small cubes in quartz

in Table 6, were taken. Number, position, temperature and occurrence of each specimen are given as by Smith. Each specimen was mounted in a cold-setting plastic at room temperature and pressure, and each was polished using the procedure outlined in Part 2, again ensuring that there was no elevation of temperature and a minimum of distortion during both mounting and polishing. In every case, for both "high" and "low" temperature material, the pyrite was distinctly anisotropic.

From this experimental and microscopic evidence it seems reasonable to conclude that the optical anisotropism of pyrite is not generally irreversibly affected by heating—to temperatures up to 550–570°C range—and that previously there has been some factor other than heat causing loss of observable anisotropism.

(2) *The effect of polishing and surface deformation.* It is of some interest to consider Smith's statement (1940, p. 91) 'It was found by accident that heat treatment of an anisotropic specimen rendered it isotropic. A fragment of the same crystal as 22 (a large cube from Brosso, Italy, R.L.S.) was ground flat and polished by hand on cloth laps . . . It showed distinct anisotropism, the colours with the nicols crossed being light blue-green to light orange-red. Seven sections were mounted in bakelite for mechanical polishing, three sections parallel to the three cube-faces, and four sections perpendicular to the body-diagonals of the cube. All were isotropic. The maximum temperature of the bakelite moulding die was 135°C and cooling to about 100°C took place in 5 minutes,' and his conclusion that heating during mounting caused loss of anisotropism.

On analysis his procedure and result present two possibilities; that disappearance of optical anisotropism has been caused by:

(a) the increase in temperature suffered by the pyrite in the bakelite press, or

(b) some deformatory action in the second polishing process avoided in the first.

As the present writer's first observations of general and pronounced anisotropism were made only after a change in polishing technique, it naturally occurred to him that the previous isotropic appearance of pyrite might have been due to surface deformation produced by other polishing methods. In other words, Smith had been presented with two possibilities—deformation during mounting or during polishing—and had assumed the first when the second may have been the case. This idea was then tested.

(a) Repolishing of material already polished on metal laps—Pyrite-bearing material earlier polished by the writer on the lead laps of the Mineragraphic Section, C.S.I.R.O., Melbourne,³ were carefully re-

³By courtesy of Dr. A. B. Edwards.

examined and the pyrite found to be isotropic. The polish was then removed by cutting back gently with American Optical Co. 304½ emery on glass, and the pyrite repolished using the new method. All material immediately appeared anisotropic. The same specimens were then repolished on lead laps at the University of Sydney, and when re-examined were isotropic. It was thus found that the *apparent* optical properties—for these specimens at least—could be changed at will simply by changing the polishing method. A small amount of confirmatory work on North American material has been carried out at the University of Western Ontario, with similar results. Material from Montgay Township, Quebec, earlier polished on cloth laps at Queen's University and found to be anisotropic, then polished on lead laps at the Canadian Geological Survey laboratories and found to be isotropic, was repolished by the writer and rendered anisotropic again. Further material (all kindly lent to the writer by Professor Hawley of Queen's University) from Teck-Hughes, Kerr-Addison and Macassa Mines, previously mounted in bakelite and polished on metal and high-speed cloth laps was found to be just slightly doubly refracting. When repolished by the writer anisotropism became quite prominent, and the normal colours and relationships displayed.

It was thus clear from these experiments that it was some feature of the polished surface, rather than variation in the body of the mineral, that was causing differences in optical behaviour.

(b) *Testing of surface effects*—The conclusion most easily seized (and in fact the one now finally adopted) was that earlier polishing methods had been deforming the mineral surfaces, making these *apparently* isotropic, and that the more recent method, in causing a minimum of deformation, revealed the true anisotropism. On the other hand it has been pointed out by Perryman & Lack (1951) that polarisation effects in cubic metals (such as aluminum) can be produced by deeply etching a polished surface—i.e. that surface contour, and hence polishing artifacts, may produce anomalous double refraction. Whether or not polishing is producing such effects can be tested by depositing (from vapour in a vacuum chamber) a thin film of silver over the surface in question. Such a film should be just thick enough to prevent transmission of normally incident light, but sufficiently thin to take up the surface contour of the surface on which it is deposited. Where polishing is not at fault, the silver film should of course be isotropic.

Several polished surfaces of apparently anisotropic pyrite were finely coated in this way. All were rendered isotropic, indicating that double refraction was produced by the mineral itself and not by surface defect.

(3) *The effect of variation in composition.* The now established fact

that pyrite is doubly refracting is clearly difficult to reconcile, at first sight anyway, with its accepted cubic symmetry.

The most common explanation of anisotropism in pyrite is that it is due to lattice strain caused by the presence of impurities—notably arsenic. Smith (1942) has suggested that variation in the properties of pyrite may be a function of two variables—composition and secondary crystal structure. He has shown that pyrite generally does not conform to the ideal composition FeS_2 and that variation in composition is always in the direction of sulphur deficiency within the limits $\text{FeS}_{2.0}$ – $\text{FeS}_{1.96}$. He suggests that iron takes the place of some sulphur in these crystals, and that this affects lattice dimensions and probably also symmetry. He suggests too that iron replacing sulphur in sulphur-deficient pyrite may be in regular positions, giving optically and electrically anisotropic crystals, or it may be in irregular positions, giving an optically isotropic variety.

Careful determinations of the trace element composition of the six North American pyrites already referred to have been made. Cobalt, nickel, molybdenum, titanium, vanadium and chromium have been estimated by careful quantitative spectrographic methods, and arsenic, antimony, bismuth, copper, zinc, lead and silver by qualitative spectrography. The results are shown in Table 7.

Copper, zinc and lead are more or less ignored as it is well-known that it is almost impossible to obtain pyrite without a few inclusions of their common sulphides. The highest cobalt and nickel figures are of the order of 0.006% and so these metals cannot be regarded as significant; those for chromium, vanadium and molybdenum are even lower. An interesting feature of the qualitative determinations is that in specimens 2 and 5 no arsenic whatever could be detected, and in all the others it was very low—a feature commented upon by Smith for some of his pyrites. It is assumed for the time being⁴ that the iron and sulphur figures are in the range obtained by Smith.

The results show clearly that arsenic is not the cause of anisotropism in at least two of the specimens, and its occurrence in the other four is quite negligible. The other elements too are present in such minute amounts that the possibility that they are causing significant lattice distortion cannot be seriously entertained. It seems that the deficiency of sulphur, as pointed out by Smith, is the only possible significant feature. A point that should be recognised, however, in any consideration of possible effects of composition *variation* is that the double refraction

⁴Accurate sulphur and iron determinations for these pyrites are being carried out, but owing to delays had not been completed in time for inclusion in this paper. The work is continuing, however.

TABLE 7. Spectrographic determinations of trace elements in the six North American pyrites listed in Table 5. Quantitative determinations are each the average of five burns. For qualitative: vs = very strong; str = strong; m = medium; w = weak; tr = trace; ftr = faint trace; nd = not detected. Analyst: J. G. MacDonald, Queen's University.

Element	Co	Ni	Cr	Mo	V	As	As	As
Wavelength	3433.0	3414.7	4254.3	3170.3	3185.4	2281.1	2349.8	2860.4
Sensitivity	0.002%	0.001%	0.0008%	0.0015%	0.004%	0.003%	0.01%	0.1%
Horne Mine (No. WO-1745)	0.057%	≤0.001%	<0.001%	0.0042%	<0.004%	nd	nd	nd
Flin Flon (No. WO-617)	0.009	<0.001	<0.001	0.0035	0.0056	tr	tr	w
Butte (WO-1786)	0.005	≤0.001	<0.001	0.0039	<0.004	w	m	m
Helen (No. 57)	0.005	0.015	<0.001	0.0022	0.0042	nd	nd	nd
Thunder Bay (No. 58)	0.006	0.0066	<0.001	0.0043	0.0062	tr	w	w
No Locality (No. 59)	0.007	0.0047	0.008	<0.002	0.0048	tr	tr	tr

Element	Pb	Cu	Zn	Sn	Ag	Sb	Bi
Wavelength	2833.0	3247.5	3345.0	2839.9	3280.6	2670.6	3067.7
Sensitivity	0.001%	0.0001%	0.01%	0.001%	0.001%	—	0.0003%
Horne Mine (No. WO-1745)	tr	vs	str.	nd	str.	nd	nd
Flin Flon (No. WO-617)	str.	vs	str.	nd	str.	ftr.	nd
Butte (WO-1786)	nd	vs	nd	m	nd	tr	nd
Helen (No. 57)	tr	m	nd	nd	nd	nd	nd
Thunder Bay (No. 58)	str.	str.	tr	tr	str.	nd	tr
No Locality (No. 59)	w	m	nd	tr	m	nd	str.

has been found to be very uniform over the whole of the wide range of material examined. This alone clearly throws extreme doubt on the possibility that iron : sulphur ratios or trace element assemblages could be significant.

Discussion and Conclusions

It is now considered established that:

- (1) Pyrite is in general optically anisotropic.

- (2) Double refraction is produced by any plane other than the octahedral.
- (3) Anisotropism is not irreversibly affected by heat up to 570°C at atmospheric pressure.
- (4) Pyrite *may* be rendered superficially isotropic by surface deformation during polishing.
- (5) Anisotropism is not necessarily due to the presence of arsenic or other "trace" impurities.

The reason for the apparent incompatibility of the findings of Smith and of the present writer concerning the behaviour of the optical anisotropism of the pyrite under heat treatment is now clear; differences are attributable quite simply to earlier limitations of technique. On the other hand as it is now evident that there is no order-disorder transformation at 135°C, the suggestion that the optical nature of the pyrite might be used as a further geothermometer must be discounted. This is emphasized by the writer's observations on material, all of which is doubly refracting, that Smith himself has measured for conductivity and stated to be of high temperature origin.

The continuity of the optical anisotropism fits with Smith's determinations of electrical and thermal properties. In view of his findings that specific resistance varied (1) with different crystallographic orientation and (2) with variation in the Fe : S ratio, and the fact that temperature-resistivity curves of crystalline solids frequently show anomalies at transition points (Kittel, 1953) it might have been expected that an order-disorder change sufficient to cause a pronounced change in optical properties would also produce a fairly sharp discontinuity in the conductivity curves. Smith's text figure (1942, p. 4, fig. 1) however, shows a remarkably smooth pair of curves with no sign of a break in the 135°C area. In view of this, any pronounced change of lattice arrangement near this temperature would have seemed anomalous.

The suggestion that the pyrite might be trigonal, although apparently fitting the electrical and thermal properties, is not supported by the optical data. As far as can be determined *all* octahedral faces are isotropic which clearly is not compatible with the trigonal uniaxial symmetry. The idea that regular replacement of some sulphur atoms by iron leads to lattice distortion and the development of trigonal symmetry, which in turn guides the development of lineage structures and the non-isometric symmetry of electrical and thermal properties is quite an elegant one, but it allows non-double refraction along only one body diagonal of the cube. If trigonal symmetry did hold the other three pairs of octohedral faces, being essentially parallel to the optic axis, should show maximum double refraction—which they unquestionably do not.

It seems that an explanation may lie in the low symmetry of the

pyrite structure, and that the double refraction might have been predicted on theoretical grounds. It is known that the crystallographic axes, although yielding four-fold *d:imensional* symmetry are, when structure is considered, only two-fold. Such a rotational symmetry would seem to fit, and may possibly explain, the observed features of double refraction, wherein similar colours appear twice in every 360° rotation and in constant relationship to cube edges. By the same reasoning other planes such as 110, 210 etc. should also be expected to produce double refraction. Octahedral planes, on the other hand, normal to three-fold axes of symmetry would not be expected to show the double refraction features of the other orientations.

That lineage structures could cause *uniform* double refraction over a whole crystal face (up to 4.0 sq. cms. in area), that they could cause the same intensity and order of double refraction on all faces of an individual cube, and that they could be the cause of essentially the same intensity and order of double refraction over the whole of a large body of samples seems unlikely. Optical properties are dictated by the structure and packing of the units of a crystal, rather than by the habit of large scale growth of aggregates of units. A lineage structure is, comparatively speaking, a coarse feature of growth and leads to the development of fairly large-scale imperfections and gaps in the body of a crystal as a whole. It does not seem likely that so coarse and so *variable* a structure could produce so uniform and so constant an effect on so delicate a phenomenon as the rotation of incident light by the few upper layers of a crystal. On the other hand density and continuity of crystal "branches" and their degree of lateral contact would undoubtedly affect conductivity, and the lineage structure idea seems a good explanation of the temperature-conductivity relationships.

Thus it is considered that the optical anisotropism and the electrical-thermal anisotropism of pyrite are essentially unrelated. The former results from the basic crystal structure of the pyrite, whereas the latter results from the grosser structures in which the former becomes involved. The first feature is very nearly a constant, the second highly variable. Thus the optical properties of pyrite are ordered and constant, whereas the electrical and thermal properties show only a rough order within an individual crystal and an enormous variation over a large range of material.

Acknowledgments

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PART 2 - THE POLISHING METHOD

ABSTRACT

It has been shown in Part 1 of this paper that pyrite is generally optically anisotropic, but that double refraction may be modified or destroyed by polishing deformation.

In this section the nature of such deformation in metals and minerals is considered, and the disadvantages of current mineralographic techniques discussed. An efficient method of mineral polishing, based on the use of a wax-abrasive mixture and diamond impregnated napless cloths, and capable of producing a high polish with little relief and low surface deformation, is then described. Several microphotographs indicating the quality of results are shown, and some implications of the improved method suggested.

Introduction

Although until now all that has been required of a polishing process in mineralography is that it should yield a smooth highly reflecting surface, it has been shown in Part 1 of this paper that requirements are in fact two-fold. A fine, highly reflecting finish is certainly—and obviously

—required, but it is also important that the surface yielded should be as representative of the underlying mineral structure as possible. It appears that in work done to date observations have been made on variably—and sometimes badly—deformed versions of minerals, rather than on the natural minerals themselves.

It is the purpose of this section of the paper to consider the nature of these deformed layers, the mechanism of their formation and ways in which their excessive development may be avoided. Reference is also made to some preliminary results obtained in the polishing of cubic minerals other than pyrite, and some implications of polishing in hardness and reflectivity determinations.

Studies in Metallographic Polishing

In considering the effects of polishing on minerals, studies of metal surfaces, although not strictly analogous, are of interest.

Sir George Beilby (1921) in his monumental work on the nature of polished metal (and other) surfaces, came to the conclusion that fine polishing was essentially a process of flowage, in which a thin film, 500 to 1,000 $\mu\mu$ thick, was rendered mobile. This mobile layer he considered to behave as a liquid and to be subject to surface tension effects, having over small time intervals, sufficient mobility to smear over and fill imperfections such as pits, scratches, and bubbles in the surface being polished. The surface film “retains its mobility for an instant, and before solidification is smoothed over by the action of surface tension” (Beilby, 1921, p. 114). On becoming rigid, this layer supposedly took on an amorphous structure and formed an essentially “vitreous” layer over the body of the metal. This has since been referred to as the “Beilby layer”.

N. K. Adam (1927) suggested that particles, possibly down to molecular size, were lifted momentarily from surfaces being polished and then redistributed, building up an even amorphous layer. Bowden & Hughes (1937) following ideas put forward earlier by Macauley (1926, 1927, 1929–32) found evidence that surface temperatures at the point of contact might, under many conditions of sliding, be sufficiently high to cause real melting of the metal, and that surface flow, polish and formation of the Beilby layer readily occurred on metals, crystals and glasses provided that the melting point of the polishing abrasive was higher than that of the substances being polished. They concluded that:

(1) The process of polishing is greatly influenced by the relative melting points of abrasive and solid.

(2) Relative hardness is comparatively unimportant.

(3) Surface flow is brought about by intense local heating of the surface irregularities to the melting or softening point.

(4) The molten or softened solid flows or is smeared over the surface, and very quickly solidifies to form the polished Beilby layer.

H. Raether (1947) in a paper of some significance gives an account of electron and x -ray diffraction and electron microscope studies of surfaces polished mechanically on the one hand and electrolytically on the other. By using both low angle reflection and transmission (peel) methods, Raether was able to study with considerable precision the behaviour of metals when submitted to cold-working, with (amongst others) the following conclusions:

(1) Cold working does in fact modify the surface layers, with disruption of the original metal structure.

(2) A finely recrystallised, as distinct from amorphous, surface zone is produced—i.e. cold-working does not produce a semi-liquid state, but fine recrystallisation (Fig. 1, after Raether, 1947).

(3) Deformation extends to a depth of the order of 10μ , with extremely fine recrystallisation to a depth of the order of 10 \AA , depending on the variables of treatment and original material.

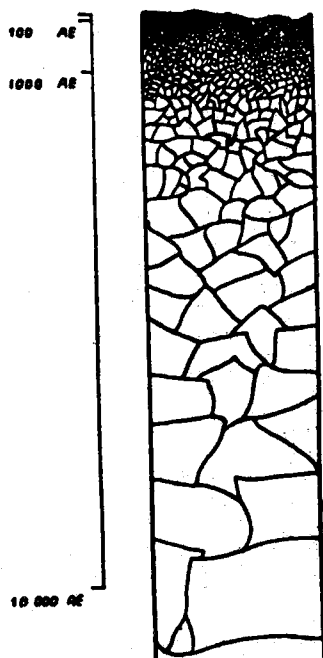


FIG. 1. Schematic representation of the upper layers of burnished copper, showing the very fine recrystallisation developed. From H. Raether, *Métaux et Corrosion*, 1947.

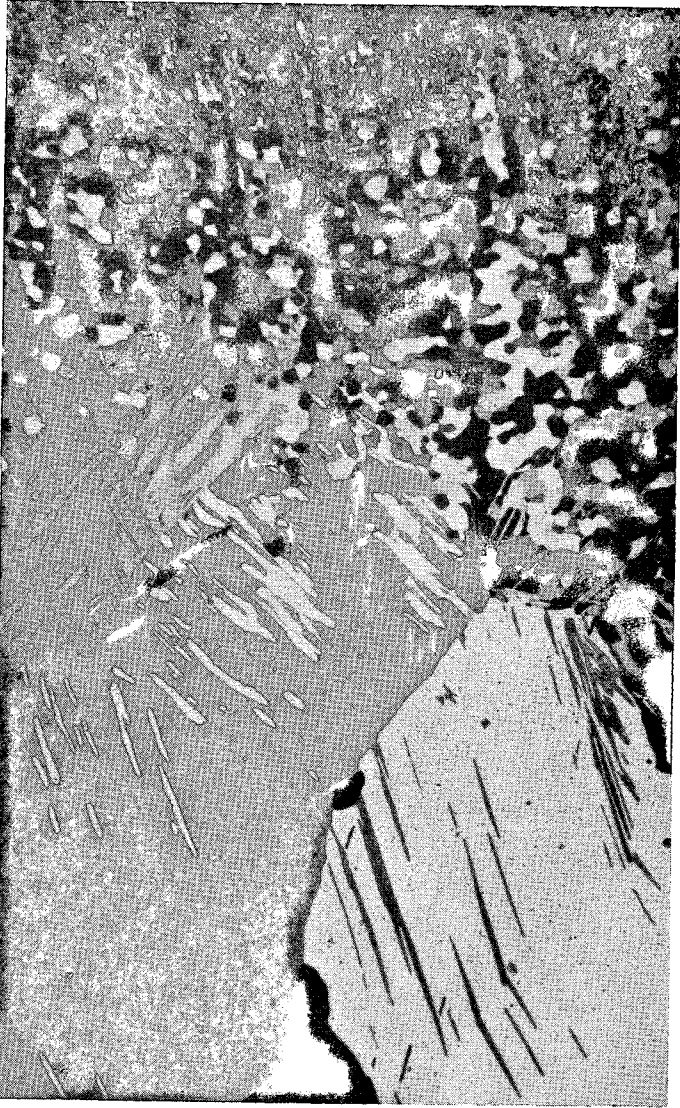


FIG. 2. Taper section of abraded zinc, showing fine recrystallisation and secondary twinning. Taper ratio, 10:1; polarised light, x 475. Photograph by L. E. Samuels.

(4) Polishing produces changes in the properties of the surface layers of metals. Incorporation of oxygen atoms causes an increase in resistance to corrosion (etching) which is not developed during electrolytic polishing. More importantly, cold-hardening is produced on a micro-scale, the depth of this varying in direct proportion to the duration and intensity of polishing and the softness of the metal. Similar treatment over five minutes produced a hardened layer 4μ thick in copper and about 80μ thick in aluminium. Hardening apparently goes deeper than the artificial recrystallisation.

(5) Because of a surplus of potential energy, the finely recrystallised material has a tendency to coarsen, such re-aggregation being a function of time and ageing temperature.

(6) Ionic crystals differ from metals in that they are brittle, not malleable. Hence degree of deformation is a function of hardness. Where deformation is produced this is by the tilting of crystal units and by the formation of a layer of disorganised, pulverised crystal fragments, whose size is of the order of at least 100 \AA diameter.

L. E. Samuels (1952-53, 1954 and in press) has carried out extensive observations on polished surfaces of metals and alloys, and exhaustive experimentation in the production of scratch-free surfaces with a minimum of non-apparent deformation. He has shown (personal communication) that abrasion produces a finely recrystallised surface layer, which grades down into a subsurface layer showing pronounced deformation twins. Deeper "rays" of deformation are associated with individual larger scratches. Samuels' conclusions, which confirm those of Raether, are particularly interesting because they have been arrived at through orthodox optical studies—of very finely polished taper sections. Figure 2, of a taper section of zinc prepared by Samuels, illustrates well the development of fine recrystallisation and twinning.

Methods in Mineral Polishing

An excellent account of the earlier methods of polishing has been given by Short (1948) and most of these are now well known. Probably the greatest single step forward in ore-polishing technique has been the introduction of the "Graton-Vanderwilt" process described by Vanderwilt in 1928, in which soft metal laps were substituted for the earlier cloth. In spite of some clear advantages, however, this method has been found unsatisfactory in many ways and over the last few years continuing efforts have been made to other workers to produce a quicker—and most important—a surer method. It can be said with fair confidence that the great retarding factor in ore studies has simply been the difficulty of polishing.

Barringer (1953-54), largely following Samuels' (1952-53) metallographic technique, has described a method using napped cloth and diamond abrasives. Hallimond (1954) has put forward a method using diamond abrasives on a solid nylon disc. Very recently Sampson (1956) has suggested a method using diamond abrasives on silk bolting cloth with high speed laps.

Discussion of Mineralographic Methods

Most of the earlier polishing methods (including the Graton-Vanderwilt method) were unsatisfactory because they were incapable of *repeatedly* satisfying the first requirement of a good polish—a flat highly reflecting surface—without at least a great deal of trouble. The more modern methods, using diamonds on cloth, nylon and metal, are far more reliable, though the alternative lap types all have disadvantages, such as excessive scratching or development of relief, depending on the material used. A further disadvantage of *all* methods so far put forward is that they cause excessive deformation of the mineral surfaces, thus all failing in varying degrees to satisfy the second requirement of a good polish.

Although Vanderwilt (1928), in describing his method, carefully considered the aims and effects of polishing, he paid only passing attention to deformation, and dismissed very quickly the possibility of the development of a Beilby layer on mineral surfaces. He states (1928, p. 300) "the suggestion of a polish film is of the greatest consequence to the geologist, for it implies that the things seen under the microscope on polished surfaces of ores are not natural geological relationships, but instead merely the structures of a superficial skin produced by polishing. The amorphous film, in so far as minerals are concerned is believed by the writer not to exist . . .". Berek (1937, noted in Turner, 1945) on the other hand, recognised the effect of polishing on mineral surfaces, and some of its optical implications. Turner *et al.* (1945) assumed without question the development of a flowed layer during polishing and showed that for stibnite Berek's characteristic angle τ decreased with progressive polishing, stating "The indication is that a flowed amorphous layer builds up thicker and thicker as the polishing proceeds". Ramdohr and other European workers have been aware that polishing deformation may produce misleading effects and Wandke (1953) has shown that lead lap polishing may have quite a pronounced effect on chalcocite, digenite and covellite and the mutual relationships of these minerals.

It has been shown irrefutably in Part 1 of this paper that lead lap methods are capable of so deforming the surface of pyrite, one of the hardest of the ore minerals, that the natural double-refraction of this

mineral has generally been obliterated in polishing, and hence has barely been recognised. It is thought that this deformation takes the form of both fragmentation and recrystallisation, resulting from the combined action of drag and frictional heating. Most Vanderwilt-type machines require a fairly heavy loading and as, for the highest polish, lubricant is normally reduced to a minimum, frictional drag is considerable. Under these conditions it is not difficult to visualise considerable tilting and fragmentation of the surface layers. In addition, it is common knowledge that lead laps frequently heat during polishing, and in fact some are water-cooled because of this. If sufficient frictional heat is generated to warm the whole body of a large lead lap it is obvious that very considerable temperatures must be developed over the almost infinitely thin mineral-lap interface. It is submitted that these two overlapping factors of drag and heating cause mechanical distortion and recrystallisation, the two varying in intensity and proportion principally depending upon hardness, melting point and crystallographic orientation of the material being polished. Such re-organisation of the surface layers into a zone of more or less randomly oriented crystal grains reduces polarisation contrast greatly and in some cases completely.

Although potentially preferable to the lead lap methods from the point of view of surface deformation, the methods of Barringer and Sampson have a limitation in the high lap speeds suggested and the high temperatures that result. Barringer (1953-54, p. 24) states that "lap speeds do not appear critical, and at a speed of 500 rpm no undue heating takes place provided the laps are well lubricated and pressure on the specimens is not too excessive", but then notes that the higher pressures required for polishing harder minerals may lead to heating, necessitating reduction of lap speed. Sampson (1956, p. 483) mentions speeds "of 800 rpm or a little more" and states that a speed of 850 rpm and strong hand pressure will warm a section in 10 to 15 seconds too hot for comfortable handling and in such cases must be cooled by removing from the lap and running water over it. He then states "However, this procedure has given some excellent results on difficult sections." Smooth surfaces, no doubt, but the deformation in many cases must be considerable.

An Alternative Polishing Method

The following method, evolved by L. E. Samuels and the present writer in 1953, is based on the use of a wax-abrasive mixture and diamond-impregnated carrier pastes on napless cloths. As far as can be judged from micrographs published, it produces surfaces at least comparable to those of other methods and, as indicated in Part 1, causes considerably less surface deformation.

(1) *Preliminary Abrasion*

The abrasive papers used in metallography are unsuitable for mineralographic work as their rigidly fixed particles cause excessive plucking in minerals with well-developed cleavages, such as galena. In some cases, their tearing action "starts" potential cleavages for some distance below the surface of a mineral grain, making later polishing very difficult. The standard method of using a succession of silicon carbide or similar abrasives on glass and/or soft metal plates is still recommended, though it must be emphasized that very coarse abrasives should not be used even in the initial stages, as these seem to cause deep loosening—a point considered by Vanderwilt himself—just as do the fixed abrasives. It has been found quite satisfactory to commence with about 400 mesh grade on a rotating gun-metal lap, and then American Optical Company 303½ and 305 on stationary glass plates.

(2) *Intermediate Abrasion—cast abrasive-wax lap*

The purpose of this lap is to improve on the finish obtained by the normal preliminary abrasion process by producing a very flat surface and by repairing most of the abrasion damage. In providing a real intermediate step, its use greatly reduces the requirements of the subsequent polishing stages.

The lap developed for metallographic purposes (Samuels, 1952–53) has been found to be equally successful in mineralography, and it cannot be emphasized too strongly that it is one, if not the, key stage of the present process. Its value in mineralography lies in the fact that its action is intermediate between that of a fixed and that of a rolling abrasive; the bonding of the particles is sufficient to produce the high cutting rate and the flat finish characteristic of a fixed abrasive, but it is not so rigid as to cause tearing, the disadvantage noted with the abrasive papers. The limitation of the wax lap is that it can be used for one, or at most two, specimens before requiring cleaning. However, an important feature of the particular lap recommended is that it can be cleaned with great ease, simply by wiping over with cotton-wool moistened with a solvent such as benzene. The lap is used stationary.

The lap itself is made of a circular metal base upon which a slab of wax-abrasive mixture is cast. The most convenient metal is probably aluminium; a disc of about 6 in. diameter is cast and machined, and the surface then sawn to give two sets of grooves at right angles, each set parallel, with 1/2 in. centres and 1/16 in. wide and deep. Such grooves are necessary to secure the wax to the metal, and this particular pattern prevents undue cracking of the wax on solidification. Wax (microcrystalline, melting point 170° C. to 190° C.) is melted and decanted to remove impurities. Silicon carbide powder (10 to 20 μ grade) is added in stages until it is impossible to incorporate any more, at which stage it should be possible to push the stiff wax-abrasive mixture cleanly away from the side of the dish with the stirring rod. During preparation of the mixture the metal

base should be fitted with a paper dam, and pre-heated. The wax-abrasive is then distributed evenly over the base plate and allowed to flatten and slowly cool.

Such a wax-abrasive slab, of about 1/4 in. thickness, should last for 300 to 400 sections, and when worn thin is simply renewed as above.

(3) *Polishing with Diamond Abrasives*

(a) Application of abrasive—It has been shown previously (16) and is now generally accepted that diamond abrasives are most efficiently and conveniently used when dispersed in a carrier paste. Generally it has been found quite satisfactory to prepare the paste in the laboratory and the following mixture has been devised by Samuels:

The components of a batch of paste of convenient size are as follows:

Stearic acid.....	12.5 gm.
Triethanolamine.....	6.0 ml.
Water.....	25.0 ml.
Diamond abrasive.....	0.5 gm.

The stearic acid is melted and heated to 80°–90° C. The triethanolamine and most of the water are mixed and heated to the same temperature range, a small amount of wetting agent and the diamond dust are added, and the abrasive shaken into a uniform suspension. The molten stearic acid is stirred vigorously (preferably with a mechanical stirrer) and the abrasive suspension introduced rapidly. The water not used in the original suspension can then be used to wash in any abrasive remaining in the container. The mixture emulsifies immediately, but stirring should be continued until the emulsion cools and thickens. The paste can then be stored in, and dispensed from, tin-lined lead collapsible tubes.

A total of about 1½ to 2" of paste is applied to spots over the central area (about 3" diameter) of the lap and lightly rubbed in with the fingertip. Cleanliness is obviously necessary here. A few drops of kerosene should be added to lubricate.

(b) Polishing cloths—Napped cloths are unsuitable for high-quality mineralographic work because the development of severe relief between individual minerals is inevitable with their use. Hallimond appreciated this point and developed a lap using a solid nylon disc. The relief was then well within acceptable limits, but the lap appears to be relatively slow in action and produces numerous rather severe scratches.

Extensive trials have shown that a napless cloth—a heavy cotton drill—is the most suitable. Little or no relief between constituents is developed, the lap has a comparatively high cutting rate, and only minor scratches are produced. It may be used for extended periods with mineral specimens, and consequently is economical to use. An important advantage of this type of cloth is that it shows no tendency to damage any of a wide range of mineral constituents so far investigated. Damage produced during preliminary abrasion may therefore be repaired with certainty though, because of the high cutting rate of the diamond abrasives and the relatively high quality of the finish obtained on the abrasive-wax lap, this rarely takes more than about two minutes.

(c) Grades of abrasive—Qualitative tests have shown that under these particular conditions of use, diamond abrasives can be used effectively only in the 0–10 μ size range. For the first polishing a 4–8 μ grade seems the most suitable, and should be used on a low speed mechanical wheel (about 150 rpm). The major polishing operation is carried out here, and only a brief finishing treatment on a hand, or low speed, lap charged with 0–1 μ grade is then necessary to produce a finish adequate for all routine visual examinations.

Each charge of the coarser grade should be sufficient for about thirty average specimens, and the finer grade, which produces much less cutting debris, should last for rather more. One or perhaps two re-applications may be made to the soiled cloth, which should then be washed thoroughly in hot soapy water, dried, again fitted to the wheel and re-charged with abrasive.

(4) *Final Polishing*¹

For fine photomicrography, a final polish is desirable to remove the minor scratches produced on the diamond abrasive pad. It is considered that a napped cloth, such as a synthetic suede, is essential for the final polishing stage if the desired freedom from scratches is to be achieved consistently. The disadvantage of napless cloths and solid laps here is that the polishing debris accumulates on the lap surface causing scratching. In the case of napped cloths, on the other hand, the debris can migrate away from the polishing surface to lodge at the base of the nap.

It is thought that any visible scratches produced on this final pad are caused by the polishing cloth rather than by the abrasive and on this basis the following final polishing technique has been evolved. Calcined magnesium oxide (from which air has been carefully excluded) is mixed into a very thick paste with water and a drop or two of detergent and pushed through a fine sieve onto the polishing pad, such sieving treatment serving to break up the paste into a thick cream. This paste is then spread over the surface of the polishing cloth and the specimen moved lightly over it, so that the surface being polished skids over a packed bed of paste without touching the nap of the cloth. The lap itself is stationary. This technique is capable of producing surfaces which, even in the softest low-melting-point metals, appear scratch-free under phase contrast illumination.

Such treatment does produce some relief between constituents. The

¹Throughout the process, from coarsest abrasion to final polishing, hand pressure should be decreased towards the end of each stage such that the final movements approach a light wiping action. This reduces the effective particle size of the abrasive, so producing a more even gradation through the polishing process. This is important.

minor relief resulting from relatively short treatments is, however, frequently an advantage, as it increases the contrast between the different minerals. There is a limit to this, of course, and the treatment should be terminated before relief becomes serious. In view of this limit to the finishing treatment, it is of some importance to ensure that the finest possible finish is obtained on the last diamond abrasive pad.

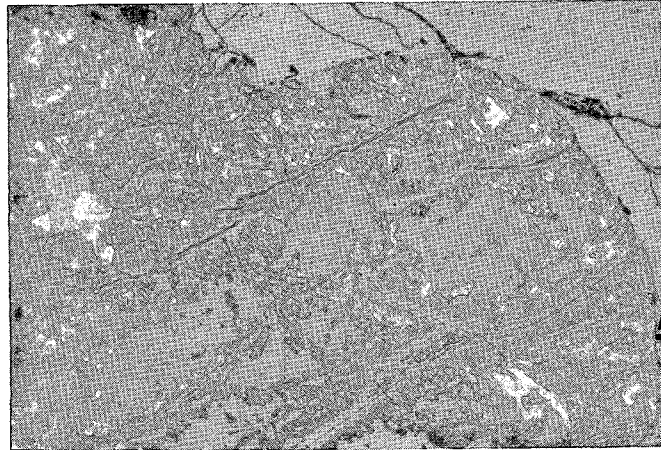
Illustration of Results

The accompanying photomicrographs have been chosen to give an indication of the quality of the average polish obtained with routine use of the method described. The minerals illustrated range in Talmage hardness (Uytenbogaardt, 1951) from F⁻ (pyrite) to B⁺ (covellite). Where possible, hard and soft minerals (e.g. pyrite and galena in fig. 4) have been shown in contact so as to give an indication of maximum relief likely and a comparison of the quality of polishes obtained. Attention is drawn to the diffuse chalcocite-covellite reaction rim about galena in figs. 10 and 11 which, although readily observed in these illustrations is barely discernible after polishing by metal lap methods.

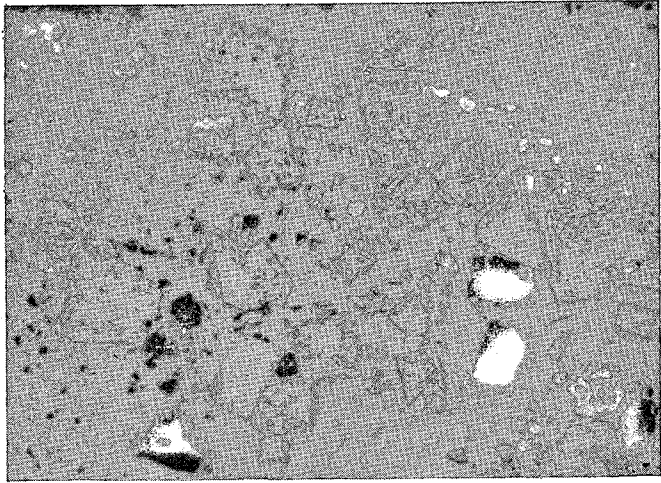
A feature of all the illustrations is the absence of major scratches. In one or two instances (e.g. figs. 9 and 11), fine scratches are discernable; had a little more time been spent on these particular surfaces, such scratches could have been eliminated. However, since the purpose of this paper has been to describe a method of routine application, it was thought best to submit photographs of polishes obtained in the normal run of work, rather than of those produced by unusually careful preparation. The average time required for preparation of the surfaces illustrated was about six to seven minutes, with a maximum of about ten. The speed with which such polishes can be obtained is clearly a most important practical advantage of the method.

Some Implications of the Method

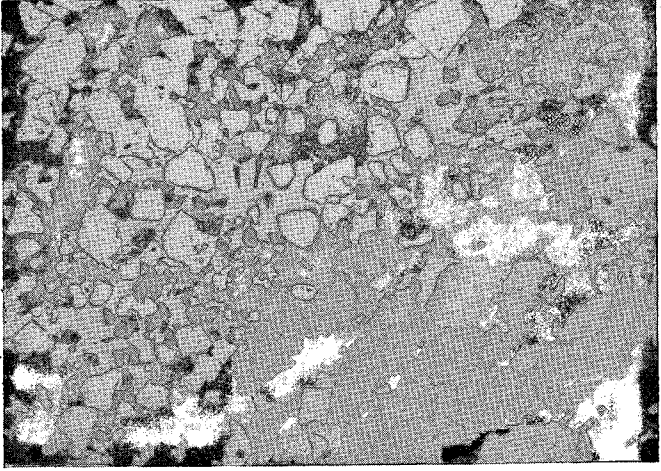
A method which permits the examination of material more closely similar to the natural substances being investigated clearly opens a field for the more accurate determination of hardness, reflectivity, polarisation and other properties which depend on the crystal structure. The more refined methods which have recently been developed to measure hardness and reflectivity are very possibly already measuring degrees of distortion and giving readings varying accordingly; in fact it is suspected that the accuracies of some of these instruments may be so much within the limits of variation—caused by deformation—of the respective properties that variations in readings are being given unmerited significance. The



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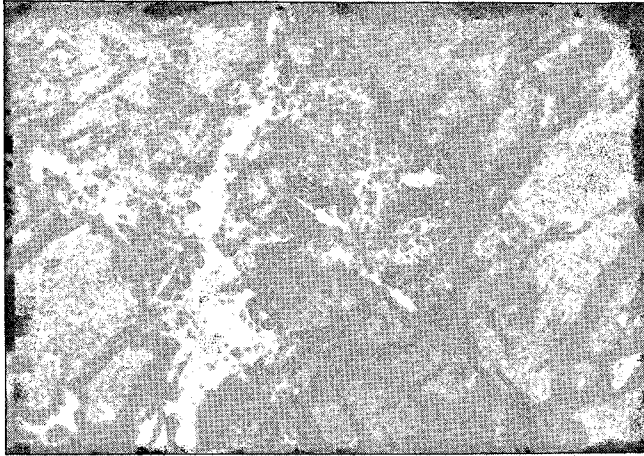


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FIG. 3. Pyrite (white, some relief), galena (off-white); tetrahedrite (light grey), sphalerite (dark grey), and gangue (black) in ore from Wiseman's Creek, N.S.W. x 45.

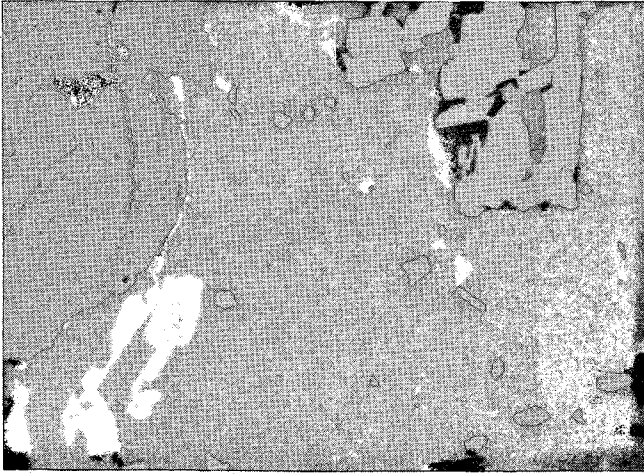
FIG. 4. Pyrite, showing extensive replacement by galena. Chalcopyrite shows up by slightly higher reflectivity towards the lower left-hand corner; sphalerite and very minor quantities of tetrahedrite are also apparent. x 45.

FIG. 5. Pyrrhotite showing alteration to marcasite along grain boundaries and fractures. A particle of sphalerite, originally within the pyrrhotite, is now isolated in marcasite. The light material is chalcopyrite. x 45.



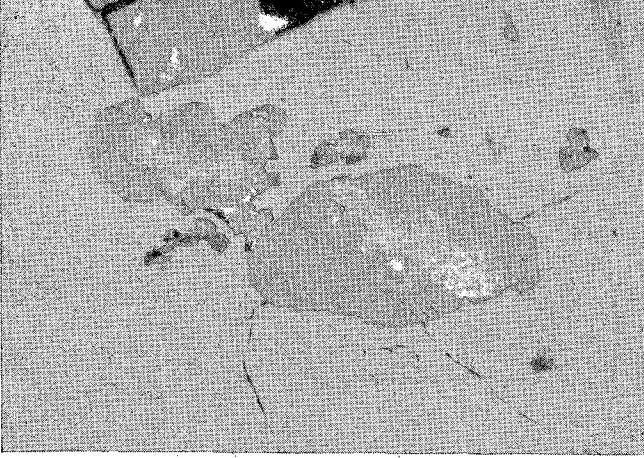
8

FIG. 8. Exsolution spindles of chalcopyrite (white) originally in bornite (dark grey) which now shows partial replacement by chalcocite (light grey). x 90.



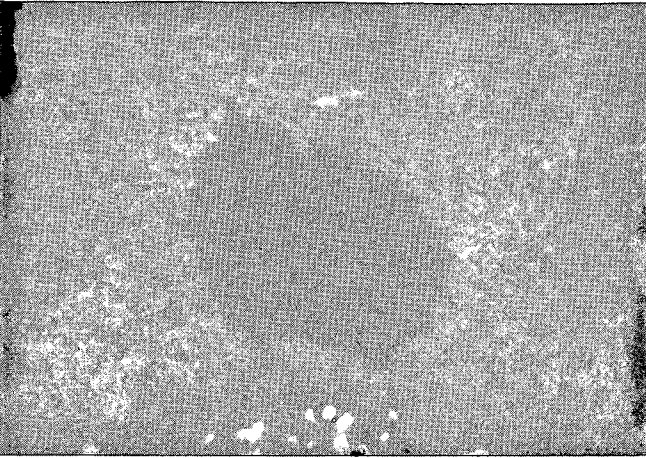
7

FIG. 7. Fine exsolution blebs of stannite within sphalerite. Large light grey mass to left is tetrahedrite; white, subidiomorphic crystals are arsenopyrite. x 450.

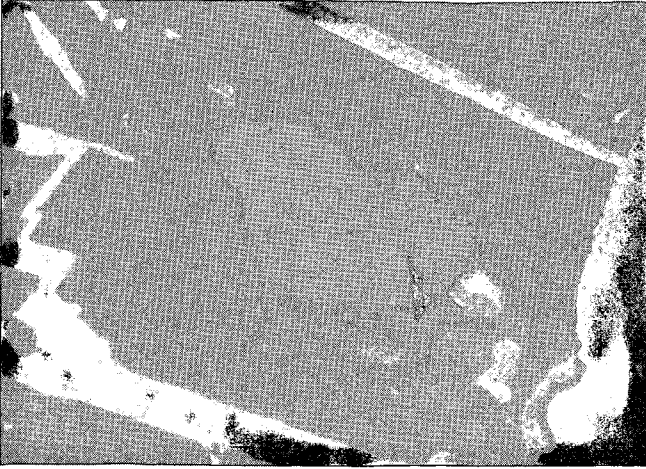


6

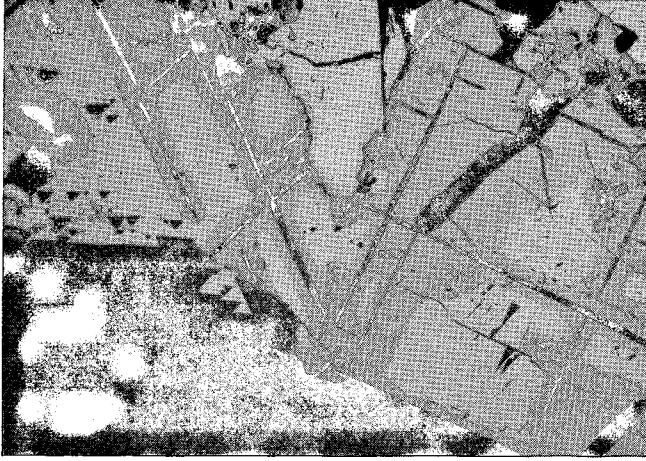
FIG. 6. Composite particles of stannite (lighter grey) and sphalerite (darker) within arsenopyrite (white background). Light grey material at top of photograph is chalcocite replacement of galena. x 225.



11



10



9

FIG. 9. Arsenopyrite (white) including small flecks of sphalerite and stannite and corroded by galena (off-white, triangular pluck marks). The galena has been replaced along grain boundaries and cleavages by chalcocite (light grey) with clear preservation of the cleavages by anglesite (very dark grey). Flecks of stannite and sphalerite, originally enclosed in galena, are now isolated in chalcocite. x 45.

FIG. 10. Chalcocite replacing galena, as in Fig. 9, at higher magnification. Note the diffuse chalcocite-covellite reaction rim separating the normal chalcocite from the galena core. x 225.

FIG. 11. Similar to Fig. 10. x 450.

work of Turner *et al.* (1945) shows clearly how surface deformation may effect, quantitatively, the polarisation effects of some minerals.

Preliminary observations now being carried out on some cubic minerals (principally cobalt and nickel sulphides and related minerals) suggests that a number of these, in addition to pyrite, may be generally doubly refracting. Anomalous anisotropism has of course been recognised in minerals such as cobaltite, gersdorffite, argentiferous galena and so on, but it seems likely that such observations will become much more frequent with the use of a finer polishing method. Apparently the film of distorted material has been just sufficient to mask the feeble double refraction produced by cubic minerals which possess a low symmetry or whose structures, owing to variations in composition, are slightly distorted. A further point of interest is that minerals such as chalcocite, already known to cause weak double refraction, show this with greater intensity and sharpness when polished with a minimum of surface deformation.

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