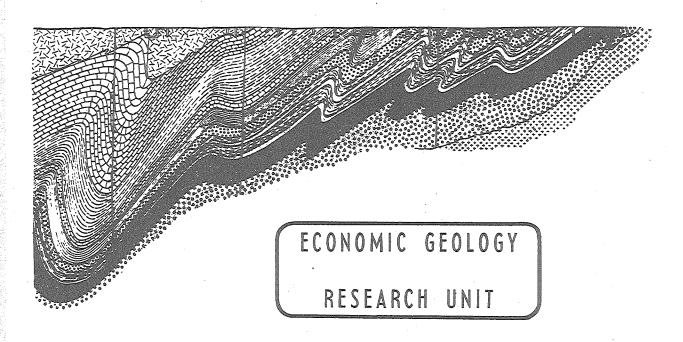


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NOTES ON TWO VARIETIES OF PYRITE FROM THE BASAL REEF OF THE FREE STATE GEDULD MINE, ORANGE FREE STATE GOLDFIELD

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ABSTRACT

Two intergrown varieties of pyrite, forming the porous buckshot pyrite in the Basal Reef of the Witwatersrand System in the Free State Goldfield were examined by various methods including that of the electron-microprobe, and the following results were obtained:

Anisotropism	Positive	Negative
Colour	Yellow with distinct reddish- brown tinge	Whitish yellow
Reflectivity at 590 m _µ	51.1%	53.2%
a _o value	5.419 Å	-
Polishing hardness	Hard	Harder
Vickers hardness (50 gram load)	1309	1622
Cobalt content	0.233%	0.049%
Nickel content	0.460%	0.114%
Arsenic content	0.877%	0.201%
Etch tests	Conc. HNO ₃ attacks strongly	Conc. HNO ₃ attacks weakly

It is pointed out that, because of the variation of its physical properties, pyrite is not an ideal reflectivity standard, although, at present, it is quite extensively used for this purpose

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CONTENTS

	Page
INTRODUCTION	1
OBSERVATIONS	1
MEASUREMENTS	2
A. Electron-Microprobe Analysis	2
B. X-Ray Data	3
C. Microhardness Determinations	4
D. Reflectivity Measurements	5
CONCLUSIONS	7
Acknowledgements	
	8
List of References	9
Key to Plates	11
Key to Figures	11

NOTES ON TWO VARIETIES OF PYRITE FROM THE BASAL REEF OF THE FREE STATE GEDULD MINE, ORANGE FREE STATE GOLDFIELD

INTRODUCTION

During a detailed microscopical investigation of the opaque minerals present in auriferous conglomerates from the Free State Geduld Mine, near Welkom, Orange Free State, two intergrown varieties of pyrite forming porous pyrite pebbles (buckshot pyrite) could be observed. These two varieties of pyrite differed, as one was isotropic and the other anisotropic. Anisotropism in pyrite has been observed by various authors, and this feature is normally regarded as anomalous.

Schneiderhöhn (23) was able to show that particularly arsenic-rich pyrites, from Upper Silesian ore-deposits, exhibited anisotropism. More recently, Uytenbogaardt (26) stated that "anisotropism (is) due to internal tension caused by some FeS surplus or As admixture". Stanton (25), however, found that the optical anisotropism of pyrite is a normal feature, but that it occurs only when the pyrite has been polished without causing a deformation of the surface layer. The anisotropism of the pyrite is, therefore, not necessarily due to its trace-element content. Similar observations were made by Arnold (1) on synthetic pyrite.

In the Witwatersrand System, Viljoen (27) mentioned anisotropic pyrite in the Main Reef Leader. However, von Rahden (19), during his study of the Ventersdorp Contact Reef, in which he examined thousands of pyrite grains, did not find any that had anisotropic properties, and he ascribed this, in terms of Stanton's explanation, to his method of polishing by making use of cloths. The close association and intergrowth in one pebble of the anisotropic and isotropic varieties of pyrite, observed by the authors, excluded the possibility that this difference in optical behaviour had been caused by the grinding or polishing process. An effort was therefore made to find the reason for the anisotropism of certain grains, in their chemical composition, and to determine quantitatively the values of reflectivity and microhardness of the two varieties of pyrite. It was found that the difference in the arsenic, cobalt, and nickel contents of the two varieties of pyrite had a marked influence upon their optical properties, their hardness, reflectivity, and their colour.

OBSERVATIONS

During the microscopic investigation of the porous pyrite nodules in the Basal Reef, it was initially observed that the pyrite occurred in peculiar fields of softer and more porous areas on the one hand, and harder and less porous areas on the other. These two areas were intimately intergrown with each other in a mosaic-like pattern, and had hypidiomorphic boundaries (Plate 1, Figure 1). Under intense illumination and oil immersion, careful observations showed a slight difference in colour. The harder variety had the normal bright whitish-yellow colour of pyrite, whereas the softer variety had a slightly lower reflectivity and a yellowish colour with a distinct reddish-brown tinge. Under crossed nicols, the softer variety showed an anisotropism comparable in intensity to that of cobaltite. Partially crossed nicols increased the anisotropic effect, and colours of slaty-grey to faint bluish-violet could be observed. At the same time, the harder variety of pyrite remained completely isotropic. Because the two varieties could just be distinguished by making use of oil immersion and at high magnifications, an effort was made to increase the difference in appearance by etching. Concentrated nitric acid applied for a period of 30 to 90 seconds showed that the anisotropic variety of pyrite was

strongly attacked and tarnished brown, whereas reaction on the isotropic variety was much slower (Plate 1, Figures 2 and 3).

A careful study of thousands of pyrite grains was carried out to investigate whether similar intergrowths of the two varieties occur in compact pyrite pebbles or secondary pyrite idioblasts. However, it was found that the two varieties could only be found in waterworn rounded porous pyrite nodules. Such pyrite nodules are present in the entire Basal Reef but are generally concentrated from near the basal contact to the upper Footwall Division. All other pyrite grains are completely isotropic, and have a colour and a reflectivity very near to that of the isotropic variety within the buckshot pyrite. The grains of buckshot pyrite have a diameter of one to five millimetres and are always larger than the much more numerous, compact pyrite pebbles, which seldom reach a diameter of two millimetres. The porous pyrite pebbles often show a skeletontype growth starting from delicate octahedral axial crosses, thus forming a very loosely knit texture. Ramdohr (20) suggested the possibility of the formation of these porous pyrite pebbles from lumps of iron silicate gels, or mixed silica limonite gels, which were slowly pyritized by the addition of hydrogen sulphide. In addition to the skeleton-type of growth-structure, numerous coliths and spherical to subspherical (of the !vererzte Bakterien'-type) structures could be observed. These can possibly be considered as relics of primitive monocellular micro-organisms rather than inorganic structures. Generally, they reach a diameter of 10 to 15 microns, but they have been observed as large as 45 microns. A more comprehensive study of these structures is in preparation by one of the authors (R.S.). The extremely delicate texture and generally weaker cataclasm of these rounded pyrite nodules, as compared with the compact pyrite pebbles, seem to indicate a short distance of transport, and possibly a younger age of formation. Besides gangue, the following minerals form inclusions in the pyrite nodules : galena, chalcopyrite, pyrrhotite, and gold. These have undoubtedly infiltrated the pyrite nodules without actual replacement. Often the nodules are coated by carbonaceous matter and secondary idiomorphic pyrite cubes, but very seldom by gold.

MEASUREMENTS

The close intergrowth of the two varieties of pyrite favoured the study of the influence of chemical composition on hardness and optical behaviour and excluded the possibility that the results could have been affected by the polishing process. All results were compared with those obtained on pyrite from Rio Marina, Elba. This pyrite from Elba was mounted and polished in identical fashion to that of the Witwatersrand samples.

A. ELECTRON-MICROPROBE ANALYSIS

The electron-microprobe is an instrument ideally suited for the determination of the composition of small sample surfaces and was used in this instance to determine the cobalt, nickel, and arsenic contents of the isotropic and anisotropic varieties of pyrite. The determination of the arsenic, nickel, and cobalt content of the sample from Elba was done spectrographically, as the concentration of these elements in the Elba sample was found to be too low for accurate determination by means of the electron-microprobe. The interest was restricted to these three elements because they are known to go into solid solution in pyrite (3, 12, 13, 14, 17). Arsenic has further been reported to cause anisotropism in pyrite (23, 26). The spectrographic analyses were carried out in the Research Laboratories of the Anglo American Corporation, and the results are presented in Table 1.

TABLE I

Results of Spectrographic Analyses of Elba Pyrite*

Nickel 80 p.p.m.

Cobalt 90 p.p.m.

Arsenic 30 p.p.m.

* Analyst : V.C.O. Schuler

The composition of the isotropic and anisotropic varieties in the buckshot pyrite was determined by means of an Applied Research Laboratories model EMX electron-microprobe. In addition to iron and sulphur, wavelength scanning revealed the presence of arsenic, nickel, and cobalt in the two varieties of pyrite. The above three elements were determined quantitatively and specture samples of the three elements, obtained from Johnson, Matthey and Co. Ltd., London, were used as reference standards. From the application of corrections described by Birks (2), results accurate to two per cent of the amounts present were obtained. The results of the quantitative determinations are tabulated in Table II.

TABLE II Results of the Quantitative Determination of Trace Elements

Type of pyrite	Arsenic %	Nickel %	Cobalt %
Isotropic variety	. 201	.114	.049
Anisotropic variety	. 877	.460	. 233

by the Electron-Microprobe on the Two Types of Pyrite

As can be seen from these results, it is obvious that the anisotropic variety of pyrite contains a considerably higher proportion of all three of these elements, and that both varieties of pyrite contain considerably more of the trace elements than the standard from Elba.

B. X-RAY DATA

Efforts to liberate enough material for an X-ray pattern of the isotropic variety of pyrite were unsuccessful because of the extremely small grain-size. X-ray patterns of the anisotropic variety and, as a comparison, those of pure Elba pyrite were recorded. The mounting technique described by Hiemstra (11) was used, and a 57.3 mm Debye Scherrer camera on a Philips PW 1008 diffraction unit was employed. Filtered cobalt Ka radiation generated at 30 kilovolts and 8 milliamperes was used. The temperature was constant at 26°C.

The pattern obtained from the anisotropic pyrite was a normal pyrite-type pattern, such as that obtained from the Elba pyrite, with the exception that the lines in the back reflection region of the anisotropic pyrite pattern were more diffuse, and suggested that there was strain in the lattice. Cell edges (a_0) were calculated for the Elba pyrite and

the anisotropic variety of pyrite, by using the method described by Henry, Lipson, and Wooster (I0). The a_0 values were calculated by carrying out a Nelson-Riley extrapolation, and are given in Table III.

TABLE III

Cell Edges of Pyrite as Determined by X-Ray Diffraction

Variety of pyrite Value of a at 26°C

Elba pyrite 5.417 + .001 A

Anisotropic pyrite 5.419 + .001A

The error of $\stackrel{+}{-}$ 0.001 $\stackrel{\wedge}{A}$, shown in Table III, is a rough indication of possible extreme lines on the Nelson-Riley extrapolation graph. Although two possible extreme lines may indicate the same a_0 value (5.418 $\stackrel{\wedge}{A}$) for both types of pyrite, it is felt that such values are unlikely, and that the difference in a_0 values for the two varieties of pyrite is significant. The larger cell edge of the anisotropic variety is to be expected because of the presence of cobalt and nickel atoms (8), but the lattice is now under strain as is also shown by the optical anisotropism.

C. MICROHARDNESS DETERMINATIONS

The microhardness of the three varieties of pyrite was determined with the aid of a Leitz Durimet Vickers hardness tester, which was positioned on a vibration-free stage. The samples were mounted in hot-setting 'lucite' under pressure and accurately levelled before measurement. Young and Millman (28) found that mounting of softer minerals at high temperature and pressure causes work hardening of the minerals and results in an anomalous increase of up to 40 per cent in the microhardness values. With harder minerals any difference caused by work hardening will be less than the probable error of the microhardness determination. The mounting process, therefore, has no effect upon the microhardness value of pyrite. All samples were polished free of relief on a Graton-Vanderwilt polishing machine. Diamond paste, and aluminium oxide slurry, and aluminium oxide paste were employed as recommended by Saager (22). The samples from Elba and the Basal Reef were prepared simultaneously, and extreme care was taken to inhibit the formation of a polishing film.

During the measurement of the microhardness, a relatively small load of 50 grms was employed. Although Young and Millman (26) recommended a load of 100 grms for minerals harder than HV 600, this load was not used because it was necessary to avoid fracturing of the small grains and to prevent the formation of large indents with diameters exceeding the grain size. Crack formation and an indent size that is too large in comparison with the grain, will obviously give unreliable results. Owing to the small diameter of the indents, and the optical error of their measurement, the microhardness values are less accurate than could normally be expected. The indentation time for all measurements was kept constant at 25 seconds, as Mitsche and Onitsch (15) have shown that the size of the indent increases with the contact time. At the same time, the distance between indents was kept as large as possible to avoid mutual interference effects. Three separate measurements were made in each variety of pyrite, and in measurements of the Elba pyrite the (111) plane was employed. Each diagonal of the indent was measured six times. All observed indents were free of cracks and showed straight edges. The results of the measurements are shown in Table IV.

TABLE IV

Results of Microhardness Measurements on the Three Varieties of Pyrite*

Variety of pyrite Value of HV

Elba pyrite 1556

Isotropic pyrite 1622

Anisotropic pyrite 1309

* Load 50 grams
Contact time 25 seconds

The microscopic observation of relief was, therefore, clearly underlined by the greater microhardness of the isotropic variety of pyrite. There is thus a clear reduction in microhardness with increase in the concentration of cobalt, nickel, and arsenic. This relationship does, however, not seem to be a very simple one as the microhardness of the Elba pyrite, which has the smallest concentration of the three trace elements, lies somewhere between those of the isotropic and anisotropic variety.

It is well known, however, that the microhardness of ore minerals can be strongly affected by the crystallographic orientation of the measured mineral, and that even optically isotropic minerals can show a distinct hardness anisotropism. Ramdohr (21), for example, mentions that the (100) face of pyrite obtains a better polish although it is harder than the (111) face. The observed difference in hardness is, therefore, not only dependent on the chemical variation, but is also affected by the unknown crystallographic orientation of the two studied varieties of pyrite. A comprehensive investigation of the variation of microhardness in relation to the orientation has only been carried out for a few minerals up to the present time.

In compact pyrite pebbles (in the same polished section as, and adjacent to, the two studied pyrite varieties), which consist of a number of pyrite individuals of different orientations (as revealed by etching), no relief was observed. In the buckshot pyrite containing the two studied varieties, a distinct relief has, however, been noticed. It is therefore felt, that the effect of chemical composition upon hardness is far more pronounced than the effect of crystallographic orientation.

Bowie and Taylor (4) gave the range of 1027 to 1240 HV for pyrite at a load of 100 grms, whereas Young and Millman (28) state the range to be 1186 to 1838. Nakhla (16), as well as Young and Millman (28), did point out that the observed microhardness was dependent on the load, and that microhardness decreases with an increasing load. According to the work of Young and Millman (28), the increase in microhardness when a load of 50 grms instead of 100 grms is used, lies between 6 and 27 per cent for hard minerals. It is obvious that the values given by the authors are too high. The reliability of their absolute values is, therefore, open to discussion but it is clear that, between the two varieties of pyrite examined, there exists a difference in hardness of the order of 25 per cent, and that this difference can thus be explained as being almost exclusively due to the difference in arsenic, nickel, and cobalt content of the two varieties.

D. REFLECTIVITY MEASUREMENTS

The determination of the reflectivities was carried out with the aid of a Leitz Dialux-Pol microscope fitted with an MPE photometer. A photomultiplier tube of the type

VFS9A manufactured by Fernseh G.M.B.H. was employed as a photocell whose spectral sensitivity is plotted in Figure 1. Dielectric filters served as monochromators, and their maximum transmission is as follows: 403 m μ , 500 m μ , 590 m μ , 626 m μ , and 649 m μ . According to Leitz specifications their half-widths are less than 25 m μ , so that even at the relatively steep portion of the pyrite dispersion curves, accurate values could be obtained. The diameter of the measured surface was 13 microns. Corrections for the effects of scattered light and reflections occurring in the inner wall of the microscope tube as well as in the objective lens and in the illuminating prism were made according to the method of Bowie and Henry (5). Naturally, care was exercised that the measured surfaces were freshly buffed and accurately positioned at right angles to the microscope axis. Each pyrite variety was measured ten times in different localities, and the photometer was checked and recalibrated when necessary between each measurement with the aid of standards (Table V).

TABLE V

Results of the Reflectivity Measurements

Wavelength	Elba pyrite	Anisotropic pyrite	Isotropic pyrite
403 mu	38.1	33.9	35.4
435 $m\mu$	41.5	36.8	39.8
500 m _µ	50.4	44.7	49.5
550 m_{μ}	53.3	49.0	51.9
590 m_{μ}	54.4	51.1	53.2
626 mµ	54.8	51.3	53.5
649 m_{μ}	53.3	51.9	53.7

The standard deviation of the ten measurements carried out on the anisotropic variety of pyrite was equal to $\frac{1}{2}$ 0.4 per cent. This is in good agreement with the statement of Piller and von Gehlen (18), who claim that, even under optimum conditions, the relative error is between 0.5 and 1 per cent. Thus, in the case of pyrite, an absolute error of 0.3 to 0.6 per cent is to be expected. Once again the (111) plane of Elba pyrite was employed as standard for which the dispersion curve was determined by Singh (24). Singh did not employ the identical pyrite crystal, but the iron content of both pyrites is very near in value, i.e. 46.63 per cent for the pyrite employed by Singh (24) and 46.78 per cent for pyrite used by the authors. In addition to this, the trace element content of the Elba pyrite is very low, so that the deviation of both varieties of pyrite probably lies below the limit of accuracy of the method employed. The standards of the National Physical Laboratory, London, were not available in South Africa at the time when these measurements were carried out.

The two dispersion curves obtained on pyrite from the Basal Reef were in good agreement with the profiles obtained by other authors. The curves have a relatively flat maximum in the yellow to red spectral region, and descend fairly steeply towards the blue end of the spectrum. The Elba pyrite, whose arsenic, cobalt, and nickel content is the lowest of the three types of pyrite investigated, shows the highest reflectivity at all wavelengths, whereas the anisotropic pyrite, which contains the highest cobalt, arsenic, and nickel concentrations, shows the lowest reflectivity. As the different varieties of pyrite were polished under identical conditions, and, because the two varieties of pyrite from the Basal Reef were so intimately intergrown, the differences in reflectivity cannot

be attributed to the polishing, but should be sought in the difference in the chemical composition of the two types of pyrite and possibly, in their differing orientations. Again, it is felt that the effect of crystallographic orientation, compared with the effect of the cobalt, nickel, and arsenic contents, is small.

A similar observation was made by Singh (24), who measured the reflectivities of pyrite from Newfoundland, South America, and Norway, and who found that the highest reflectivity was present in pyrite with the least stoichiometric composition (Newfoundland pyrite). In his case he also did not know the orientation of the pyrite, while the trace element concentration was known to him only semiquantitatively and was in each case lower than 0.1 per cent.

The shape of the observed dispersion curves for both pyrite varieties shows clearly the subjective colour impression given by these two pyrite varieties (Figure 2). In the case of the light whitish-yellow isotropic pyrite, the curve ascends a little more sharply in the blue-green range so that the values between wavelengths 480 and 550 m_{μ} are relatively higher than in the case of the anisotropic pyrite, which has a more yellowish colour-tone with a definite reddish-brown tinge (Table VI).

TABLE VI

Proportions of the Measured Reflectivity Values for the Isotropic and Anisotropic Varieties of Pyrite at Different Wavelengths

Risotropic	Wavelength
Ranisotropic	m_{μ}
1.04	403
1.08	435
1.11	500
1.06	550
1.04	590
1.04	626
1.04	649

The possible pleochroism of the anisotropic pyrite, if present, must lie below the limit of the measuring sensitivity of the instrument, and it could therefore not be determined.

CONCLUSIONS

The results of the cobalt, nickel, and arsenic determinations on two optically different pyrite varieties from the Basal Reef of the Witwatersrand System in the Free State Goldfield, showed distinctly that the observed intensity of the anisotropism of pyrite varies strongly with the content of these three trace elements. In addition to this, it was found that reflectivity and microhardness values of the two varieties of pyrite were also dependent upon their content of these three elements, in such a fashion, that the reflectivity

and microhardness were found to decrease with increasing arsenic, cobalt, and nickel content. The subjective colour impression of the two pyrite varieties differs somewhat, as is to be expected from the different shapes of their dispersion curves. During the above investigation it was not possible to determine which of the three trace elements has the strongest, or possibly the only, influence upon the varying physical properties of the pyrite varieties and it is felt that here is an interesting field for fruitful further research.

The strong interdependence of chemical composition and reflectivity, and the related differences in the shape of the dispersions curves rule out the use of pyrite as a reliable universal standard for reflectivity determinations, as the cobalt, arsenic, and nickel contents of this mineral are generally unknown. The obtained microhardness values show that, with such determinations, the contents of arsenic, nickel, and cobalt play at least as important a role as the crystallographic orientation. The observed values of microhardness are therefore, not reproducible without the concentration of these trace elements being stated. Even under identical conditions of measurement and orientation, large differences in the microhardness of pyrite from different localities are to be expected. This is actually borne out by published data.

Lastly, the genetic significance of the cobalt and nickel contents of the two studied types of pyrite should be discussed. According to Carstens (6, 7), and Hegemann (9), pyrite of sedimentary origin contains less than 100 p.p.m. cobalt and has a cobalt to nickel ratio of less than one. Hydrothermal pyrite has, however, a cobalt content between 400 and 2400 p.p.m. and the cobalt to nickel ratio is larger than one. The obtained cobalt and nickel values of the pyrite varieties investigated in this study indicate both a sedimentary (cobalt to nickel ratio < 1) and a hydrothermal (cobalt > 100 p.p.m.) origin. The extremely delicate nature and skeleton-type growth of the porous pyrite nodules strongly indicate an 'in situ' formation and a small distance of transportation. Accordingly, a formation from Fe-rich silica gels by the addition of H_2S is proposed. The occurrence of the two pyrite varieties in mosaic-like, intimately intergrown, areas and the absence of any zonation, indicate that areas markedly differing in cobalt and nickel contents were a primary feature and have simply been retained during pyritization. The cobalt and nickel values in this case cannot thus be used as a criterion in genetic interpretations. Zimmermann (29) in his investigation of the Mount Isa ore bodies reached a similar conclusion.

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Key to Plates

Figure 1: Edge of porous buckshot pyrite grain showing intergrowth of isotropic (white) and anisotropic (light grey) pyrite. Galena (dark grey) forms small inclusions. Gangue (black). Free State Geduld Mine, Basal Reef. Reflected light, oil immersion. (X 600).

Figure 2: Intergrowth of isotropic (white) and anisotropic (light grey) pyrite prior to etching. Free State Geduld Mine, Basal Reef. Reflected light, oil immersion. (X 320).

Figure 3 : Same specimen as in Figure 2 but etched for 90 seconds with concentrated HNO3. Anisotropic variety strongly attacked. Reflected light, oil immersion. $(\times 320)$.

Key to Figures

Figure 1 : Spectral sensitivity of the photomultiplier tube employed in the reflectivity measurements.

Figure 2: Dispersion curves of three pyrite varieties, containing different amounts of cobalt, nickel, and arsenic. Elba pyrite, after Singh (24).

PLATE 1

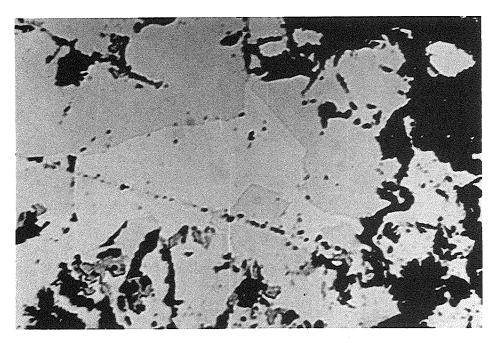
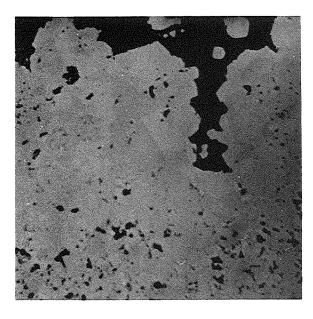


Fig. 1



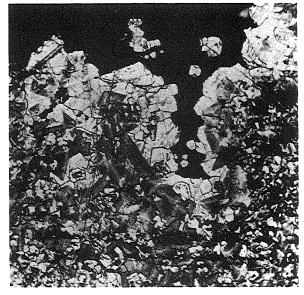
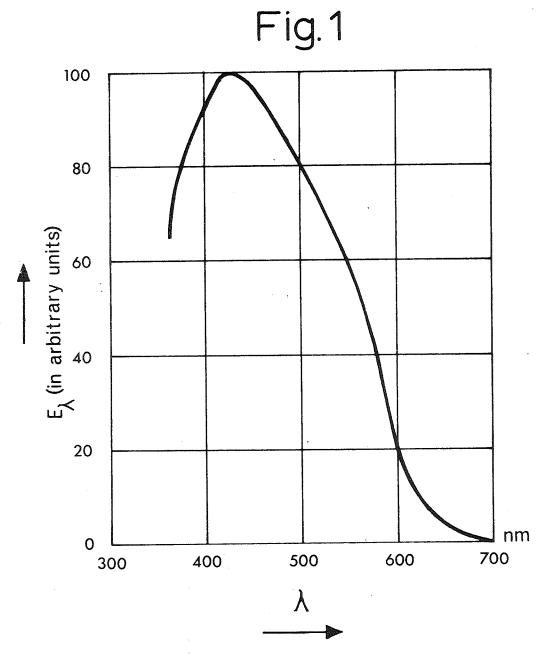
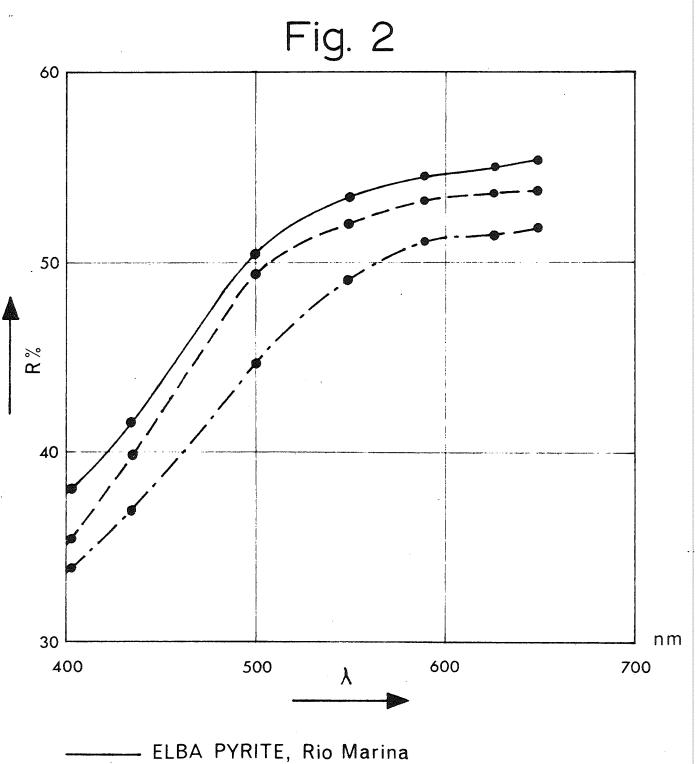


Fig. 2 Fig. 3



 E_{λ} : Spectral sensitivity of the V FS 9A photomultiplier tube



ISOTROPIC PYRITE, Basal Reef

ANISOTROPIC PYRITE, Basal Reef