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SHORTER COMMUNICATIONS

NICKEL DISTRIBUTION IN HEXAGONAL AND MONOCLINIC PYRRHOTITE

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This note describes the distribution of nickel in solid solution in pyrrhotite between the hexagonal ("nonmagnetic") and monoclinic ("magnetic") forms. Arnold (1969), Yund & Hall (1969), others have studied the Fe-S phase diagram in great detail. At least three naturally occurring phases exist in the FeS region : troilite, stoichiometric FeS ; low temperature hexagonal pyrrhotite, $Fe_{1-x}S$; and low temperature monoclinic pyrrhotite, $Fe_{1-x}S$. The magnetic susceptibility of the pyrrhotite phases has been studied by Schwarz (1968), Hayase (1963) and others. For all practical purposes the monoclinic phase is magnetic and the hexagonal phase is nonmagnetic.

Significant concentrations of nickel in solid solution occur in many pyrrhotite samples. This concentration of nickel was studied by quantitative electron probe analysis of sixty-five randomly selected grains from a composite sample representing a specific ore body from the Sudbury area. Each grain was analyzed at four randomly selected points. Each point covers an area of 4 square μm. All measurements were taken away from visible pentlandite exsolution flames.

A frequency plot (Figure 1) of the measured nickel concentrations shows a bimodal distribution. Sixteen grains yielded "high" nickel values only, twenty grains showed "low" nickel values only and twenty-nine grains gave both "high" and "low" values. Pure iron and nickel were used as standards for the metals and a homogeneous, previously analyzed grain of pyrite served as a sulphur standard. The raw data were corrected using the computer program MAGIC III. Final nickel concentrations are given in Table 1.

A polished section was etched in dilute chromic acid and microscopic examination showed the presence of two finely intergrown phases. Furthermore, as stated above many grains gave both "high" and "low" nickel

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values. The relationship of these two observations was tested by a detailed probe analysis of one of the many grains showing a fine intergrowth (Figure 2). A light repolishing removed the etch and probe analysis was performed on a selected area of 100 μm by 100 μm. The probe "burn" marks show the exact location of analyzed points. These data points were then plotted on a photograph of the etched specimen. The result is seen in Figure 3. A magnetic colloid was deposited on the grain high-lighting th magnetic properties of the two phases. This is shown in Figure 4.

The photographs show the existence of a definite correlation between such naturally occurring properties as structural form and magnetic susceptibility and the nickel content of pyrrhotite. The magnetic monoclinic pyrrhotite in this sample contains between 0.4-0.5% nickel and the hexagonal paramagnetic phase contains between 0.8-0.9% nickel.

In considering "average" nickel values in pyrrhotite therefore one must take into account the existence of two distinct pyrrhotite modifications having different nickel content.

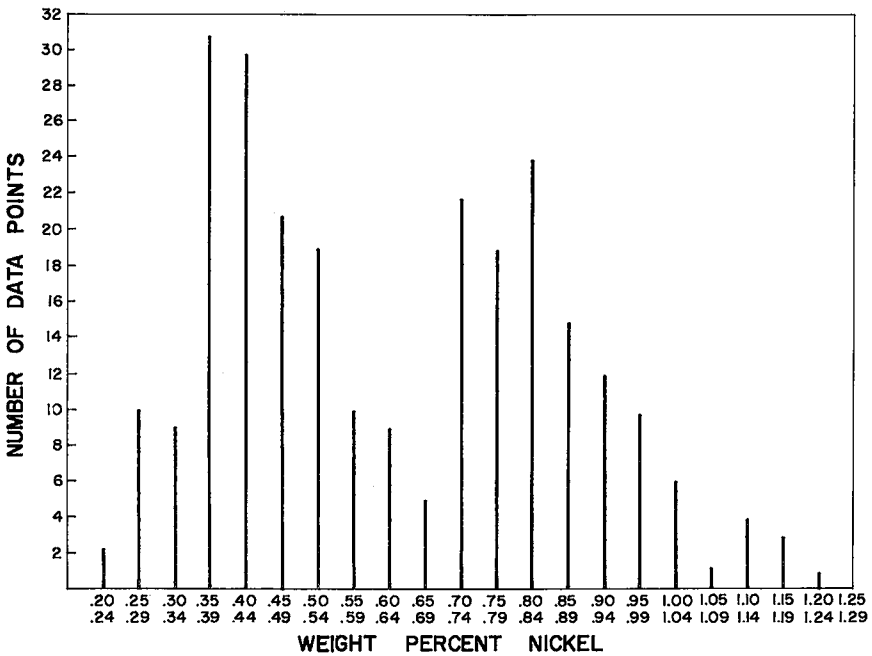


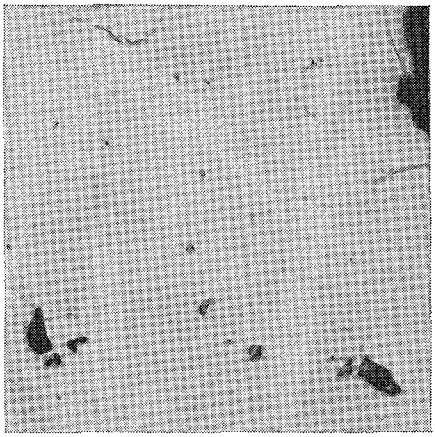
FIG. 1. A frequency plot of the measured nickel concentration in 65 randomly chosen grains. The abscisse shows the ranges in nickel concentrations and the ordinate the number of times each concentration range was encountered.

TABLE 1. WEIGHT PERCENT NICKEL IN PYRRHOTITE

Grain	% Ni	Grain	% Ni	Grain	% Ni
1	0.85	12	0.42	23	0.94
	0.78		0.54		0.86
	0.75		0.52		0.96
	0.79		0.41		0.98
2	0.79	13	0.23	24	0.49
	0.58		0.62		0.57
	0.83		0.27		0.47
	0.66		0.80		0.49
3	0.83	14	0.46	25	1.18
	0.41		0.47		1.16
	0.39		0.50		1.13
	0.47		0.47		1.20
4	1.10	15	1.06	26	0.31
	1.14		0.67		0.27
	0.91		0.28		0.32
	0.79		1.04		0.27
5	0.61	16	0.80	27	0.90
	0.46		0.72		0.85
	0.94		0.48		1.00
	0.89		0.48		0.92
6	0.97	17	0.64	28	0.28
	0.92		0.34		0.72
	0.98		0.58		0.60
	1.00		0.49		0.72
7	0.59	18	0.46	29	0.43
	0.41		0.44		0.81
	0.79		0.44		0.89
	0.65		0.37		0.35
8	0.26	19	0.38	30	0.29
	0.29		0.42		0.38
	0.26		0.44		0.54
	0.27		0.43		0.31
9	0.32	20	0.70	31	0.86
	0.32		0.71		1.01
	0.29		0.72		1.02
	0.44		0.73		1.04
10	0.40	21	0.51	32	0.74
	0.37		0.42		0.77
	0.39		0.70		0.79
	0.32		0.57		0.83
11	0.49	22	0.89	33	0.38
	0.54		0.91		0.40
	0.51		0.49		0.40
	0.86		0.55		0.37

TABLE 1 (cont'd)

Grain	% Ni	Grain	% Ni	Grain	% Ni
34	0.37	45	0.50	56	0.97
	0.42		0.51		0.87
	0.71		0.50		0.95
	0.40		0.49		0.91
35	0.78	46	0.85	57	0.51
	0.66		0.80		0.50
	0.42		0.74		0.50
	0.41		0.72		0.54
36	0.41	47	0.93	58	0.61
	0.44		0.98		0.71
	0.67		1.04		0.43
	0.83		1.06		0.63
37	0.31	48	0.50	59	0.61
	0.41		0.45		0.60
	0.80		0.52		0.56
	0.82		0.58		0.59
38	0.86	49	0.39	60	0.87
	0.61		0.47		0.86
	0.80		0.39		0.84
	0.82		0.35		0.45
39	0.79	50	0.84	61	0.78
	0.31		0.82		0.39
	0.76		0.84		0.74
	0.39		0.70		0.36
40	0.88	51	0.51	62	0.44
	0.79		0.50		0.90
	0.90		0.41		0.90
	0.81		0.38		0.56
41	0.82	52	0.39	63	0.39
	0.71		0.40		0.45
	0.82		0.39		0.45
	0.77		0.35		0.44
42	0.70	53	0.50	64	0.75
	0.73		0.47		0.75
	0.78		0.48		0.38
	0.77		0.49		0.80
43	0.74	54	0.95	65	0.36
	0.35		0.97		0.41
	0.34		0.87		0.31
	0.25		0.49		0.38
44	0.71	55	0.83		
	0.40		0.71		
	0.71		0.80		
	0.37		0.24		



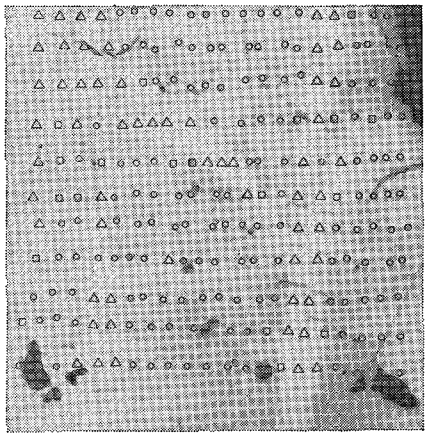


FIG. 2 (top left). Photomicrograph showing monoclinic and hexagonal pyrrhotite intergrowth. The sample was etched in dilute chromic acid for ten minutes and photographed through Nomarski interference contrast optics using an oil objective, × 600.

FIG. 3 (top right). Nickel concentration map of the pyrrhotite grain shown in Figure 2. The concentration of nickel as measured by the probe has been superimposed on a photograph of the etched grain seen in Figure 2. The correlation of the low nickel concentration with the monoclinic phase is evident. The symbols have been placed on "exactly" the positions analyzed by the probe. The symbols correspond to the following weight percent nickel: ○ = 0.8-0.9%, □ = 0.6-0.8%, △ = 0.4-0.5%. Magnification × 600.

FIG. 4 (bottom). Magnetic structure of pyrrhotite grain shown in Figures 2 and 3. A magnetic colloidal dispersion was placed on the grain and the solution allowed to evaporate. The magnetic particles adhered to the low nickel phase. Magnification × 600.

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References

ARNOLD, R.G. (1969) : Pyrrhotite phase relations below 304 ± 6°C at 1 atm. total pressure. *Econ. Geol.* **64,** 405-419.

HAYASE, K. *et al.* (1963) : On the magnetic properties of natural pyrrhotites. *Mineralogical J.* **4,** 41-56.

SCHWARZ, E.J. (1968) : Magnetic phases in natural pyrrhotite $Fe_{9.89}S$ and $Fe_{0.91}S$. *J. Geomag. Geoelec.* **20,** 67-74.

YUND, R.A. & HALL, H.T. (1969) : Hexagonal and monoclinic pyrrhotites. *Econ. Geol.* **64,** 420-423.

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ELECTRON PROBE MODIFICATIONS USING POLARIZED LIGHT FOR MINERALOGICAL GRAIN EXAMINATION

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A Philips AMR/3 electron-probe microanalyzer has been used to determine the chemical composition of individual grains in plagioclases. For this use the microanalyzer was modified to enable thin transparent mineralogical specimens to be viewed in transmitted polarized light while in position under the electron beam ; thereby bringing out the grain structure.

A sketch of the general arrangement of the specimen and optical system is shown in Figure 1. A 400 watt source of white light with a system of lenses was used to produce a collimated beam. To direct this beam through the system of polarizer, sample and analyzer, an inclined plane mirror was placed directly under the focusing piston as shown. The light source and the mirror were adjusted to obtain maximum light intensity at the eye-piece. The analyzer, which was attached to the eye-piece, could be rotated to bring the desired grain into view.

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