

MINERALOGICAL NOTES

NOTE ON THE MEASUREMENT OF PYRRHOTITE COMPOSITION IN THE PRESENCE OF BOTH HEXAGONAL AND MONOCLINIC PHASES

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Recent research on metal contents of pyrrhotite, using the *x*-ray spacing method outlined by Arnold and Reichen (1962), has resulted in some adaptations to the original method.

This method involved crushing each sample under inert liquid to avoid oxidation, and concentration by magnetic separation prior to *x*-ray diffraction. This procedure was found to be extremely tedious and crushing in air was investigated as a possible alternative. Diffraction patterns given by equivalent pyrrhotites, freshly crushed in air and under inert liquid, were compared and found to be identical. This indicates that the effects of oxidation are negligible over a short period.

The inversion of monoclinic pyrrhotite to hexagonal symmetry by heating at 600° C. was also examined. The majority of specimens investigated exhibited two reflections, about 0.35° 2θ apart, the relative intensity of these peaks varying quite considerably. The reflections were measured prior to heating and again following heating at 600° C., for 18 hours in an evacuated, sealed silica glass tube. The resulting single reflection was equivalent in position to the first of the two reflections attained prior to heating, but was of greater intensity (Fig. 1). It would appear that the double reflections represent the presence of both the hexagonal and monoclinic forms in varying proportions, the monoclinic pyrrhotite reverting to the hexagonal form on heating and rapid cooling. Thus measurement of $d(102)$ for the reflection having the lower value of 2θ when both structural types are present, should be sufficient for the determination of the composition of the sample.

Diffractometer traces were prepared using $\text{FeK}\alpha$ radiation in a Philips diffractometer. The position of the peak was found by using the technique of counting for a fixed time, most of the peaks being sufficiently intense and sharp to be readily differentiated from the background. Occasionally where peaks were not well resolved, sufficiently accurate determinations were made using a scanning speed of $\frac{1}{4}^\circ 2\theta$ per minute, time constant 4 seconds, and a high chart speed.

Forty-eight pyrrhotites from the Mt. Bischoff Mine in Tasmania were examined using *x*-ray diffraction techniques described by Arnold and Reichen. A general relationship between crystal symmetry and metal content was obtained, pyrrhotites exhibiting monoclinic symmetry being the more metal deficient phase. This substantiates the results of Grønvold

and Haraldsen (1952). Twelve pyrrhotites containing greater than 47.20 atomic per cent metals were examined and found to be exclusively hexagonal although in many cases a very low intensity scattering affect was present in place of the monoclinic peak. In more metal-deficient pyrrhotites, monoclinic symmetry is exhibited in varying proportions and in pyrrhotites containing less than 46.80 atomic per cent metals it is always present. Formation of the monoclinic phase is considered to be a rela-

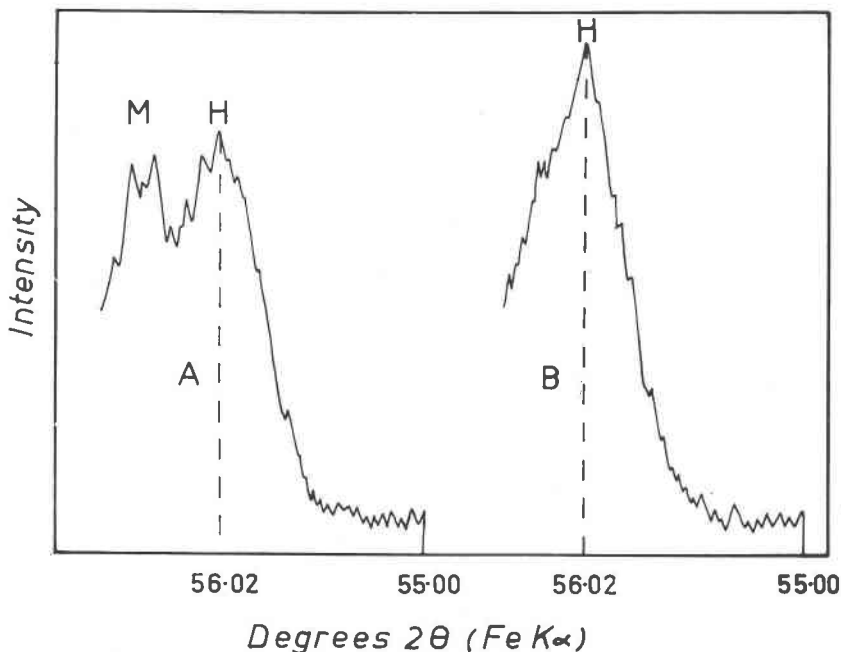


FIG. 1

tively low-temperature transformation from unstable, metal-deficient, hexagonal pyrrhotite, crystallized at high temperatures, to a super-lattice type structure as previously indicated by Grønvold and Haraldsen (1952). The high temperature of formation is indicated by low metal content (Arnold, 1962). The preservation of constant composition during this transformation is indicated by the fixed position of the hexagonal reflection, as previously indicated, and the absence of exsolution textures in polished section of pyrrhotite containing the hexagonal and monoclinic form.

Arnold and Reichen report exclusively hexagonal pyrrhotite containing as little as 46.70 atomic per cent metals, this being below the limit of its occurrence determined by the authors. This could be due to the variable

cooling histories of the pyrrhotites, because slow cooling would tend to favor the formation of monoclinic pyrrhotite, particularly for pyrrhotites with compositions near the upper limit for iron. For lesser amounts of iron it would probably appear as a distinct phase regardless of cooling history. It is hoped to verify this relationship by heating and slow cooling of natural pyrrhotites across the transformation boundary between the hexagonal and monoclinic phase.

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THE UNIT CELL AND SPACE GROUP OF STEWARTITE¹

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Tennyson (1956) reported that stewartite, $MnFe_2(OH)_2(PO_4)_2 \cdot 8H_2O$, is monoclinic with the unit cell parameters listed in Table 1. The value of the magnitude of b (60.8 Å) is exceptionally large by comparison with parameters of reduced unit cells of most inorganic substances. Accordingly, the unit cell has been reinvestigated.

Crystals of stewartite from the Palermo and Fletcher quarries, N. Grotton, N. H., were used in this investigation. The stewartite from the Palermo quarry occurs as radiating groups of small yellow crystals associated with strunzite, laueite, eosphorite, "diadochite," rockbridgeite, and siderite, while that from the Fletcher quarry occurs as aggregates of yellow crystals in cavities in rockbridgeite with hureaulite, strengite and metastrengite. In both cases the minerals are alteration products of triphylite. The identity of the stewartite was confirmed with powder photographs, the patterns corresponding closely to those described by Murdoch (1958) and Neves (1958, 1960).

The crystals from the Palermo quarry resemble those described by Tennyson from Hagendorf, and are bounded by three pinacoids. Rota-

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