

MACKINAWITE AND VALLERIITE
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ABSTRACT

Mackinawite and valleriite have been observed in the dunites and pyroxenites of the central layered series of the Muskox intrusion, Northwest Territories, Canada. New electron probe measurements and x-ray powder diffraction data are presented which compare closely with those given by earlier workers. Certain optical and physical properties that were found to be helpful in distinguishing the two minerals in polished section are tabulated. Mackinawite occurs as a replacement in pentlandite and is invariably associated with this mineral in serpentine-bearing rocks. Valleriite occurs in serpentine where it may replace secondary magnetite and, unlike mackinawite, is not usually associated with other sulfides. Mackinawite formed before valleriite and various lines of evidence suggest that both minerals formed at sub-magmatic temperatures during, and as a result of, the serpentinization process.

INTRODUCTION

Both mackinawite and valleriite have been identified in the course of a general study of sulfides and related minerals in the Muskox ultramafic intrusion (Chamberlain, in preparation). Mackinawite is a minor but widespread member of sulfide assemblages in dunites and pyroxenites in parts of the central layered series. Valleriite is observed in the same zone and appears to be less abundant than mackinawite.

The iron sulfide, mackinawite, has recently been named in a paper by Evans, *et al.* (1964) which is a follow-up of earlier work done on the sulfides of the Mackinaw mine, Washington, by Milton and Milton (1958). Natural occurrences of the same compound from Finland were described by Kouvo *et al.* (1959, 1963) and it was prepared artificially by Berner (1962). Similar material was noted by Meyer *et al.* (1958), and by earlier workers, as a corrosion product of petroleum-carrying pipe in hydrogen sulfide environments.

The composition of valleriite has been in doubt since the mineral was originally described by Blomstrand nearly 100 years ago. However, recent work by Evans *et al.* (1962, 1964) indicates that it has a formula close to CuFeS_2 . These authors believe that much of the valleriite mentioned in the literature is actually mackinawite.

It is intended that the present paper provide data that will assist in distinguishing mackinawite from valleriite as routinely observed in polished section. In addition, some information is presented on the occurrence of the two minerals in the Muskox intrusion which appears to shed light on their mode of origin.

ACKNOWLEDGMENTS

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HISTORY

The Muskox intrusion was discovered in the Coppermine River area of northern Canada in 1956 by the Canadian Nickel Company and mapped by C. H. Smith of the Geological Survey of Canada in 1959 and 1960. The preliminary results of this work are required reading for anyone interested in becoming familiar with the geology of the intrusion (Smith, 1962; Smith and Kapp, 1963). In 1963 the Geological Survey of Canada drilled the intrusion as part of Canadian studies related to the International Upper Mantle Project. Three vertical holes were drilled to a combined depth of 10,000 feet, and the results of this project are described by Findlay and Smith (1964). Samples from the drilling provided most of the specimens used in the present study.

GENERAL GEOLOGY

For present purposes, the best description of the nature and form of the Muskox intrusion is given in an abstract by Smith and Kapp (1963) as follows:

"The Muskox intrusion is a Precambrian layered pluton, 74 miles in length, which is dike-like in plan and funnel-shaped in cross section. Its internal structure is divided into four principal units—a feeder, marginal zones, a central layered series and an upper border zone. The feeder contains bronzite gabbro and picrite in zones parallel to the nearly vertical walls. The marginal zones parallel the walls of the intrusion which dip inward at angles of 23 to 57 degrees, and grade inward from bronzite gabbro at the contact through picrite and feldspathic peridotite, to peridotite and, in places, dunite. The central layered series is 6,500 feet thick and contains 34 main layers of dunite, peridotite, pyroxenites and gabbros which vary in thickness from 10 to 1,100 feet. These layers are nearly flat-lying and discordant to the marginal zones. The upper border zone is less than 200 feet thick and is characterized by an upward gradation from granophyre-bearing gabbro to granophyre."

Some of these features are illustrated in Fig. 1, a vertical east-west cross section through the intrusion.

Sulfides in the Muskox intrusion are most abundant along its margins and roof, and in a chromite-rich horizon in the upper part of the central layered series. In addition to occurring in these distinct zones, sulfides are

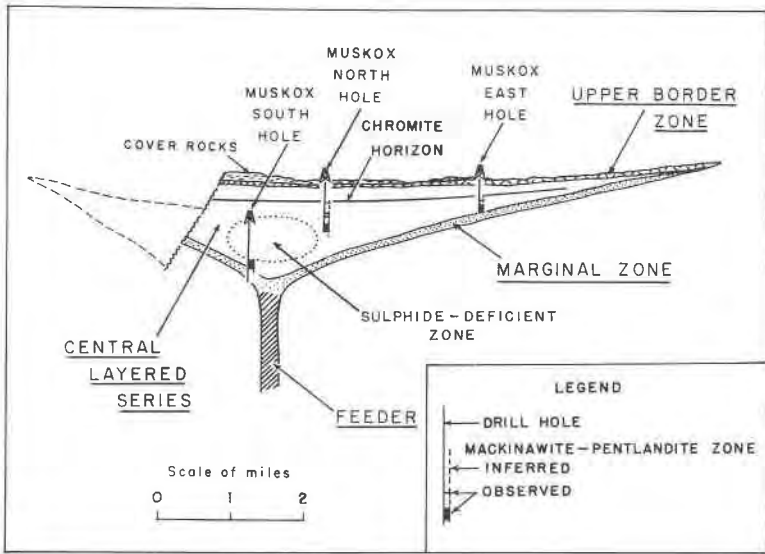


FIG. 1. Vertical cross-section through Muskox intrusion showing distribution of mackinawite-pentlandite assemblage as outlined by deep drilling.

thinly distributed through the upper part of the central layered series and upper border zone where they seldom exceed 0.1 per cent of the rock. A sulfide-deficient zone exists in the lower part of the central layered series (Fig. 1). Sulfides are common minor constituents of the Muskox feeder, and are moderately common in most of the diabase dikes of the area.

MINERALOGY

Mackinawite. Chemical analyses of mackinawite from the Outokumpu mine are given by Kouvo *et al.* (1963) in Table 1.

Electron micro-probe measurements on similar material by both Kouvo

TABLE 1. CHEMICAL ANALYSES OF MACKINAWITE¹

	Sample 1	Sample 2
Fe	55.94	56.35
Ni	8.26	8.17
Co	0.42	0.37
Cu	0.09	0.05
S	35.29	35.06

¹ Outokumpu mine, Finland (Kouvo *et al.*, 1963); corrected to 100 per cent.

et al. (1963) and Evans *et al.* (1964) are listed in Table 2, along with a measurement of one grain from the Muskox intrusion. These results indicate that the composition of the mineral is close to FeS.

The physical and optical properties of the Muskox mackinawite are summarized as follows: the mineral takes a fair polish, never without scratches and shows a well-developed cleavage in one direction. It has a Vickers hardness of 58 which makes it somewhat softer than galena, and slightly harder than native silver. This is in contrast to the Outokumpu mackinawite which is listed by Kouvo *et al.* (1963) as having a hardness "greater than that of pyrrhotite but lower than that of pentlandite rich in cobalt." The hardness of the Muskox mackinawite was measured along

TABLE 2. ELECTRON MICRO-PROBE MEASUREMENTS OF MACKINAWITE

	1	2	3	4	5	6	7
Fe	63.2±2	64.7±2	58.9±2	55.1±2	53.1±2	63 ±5	55 ±5
Ni	0.2	0.2	0.5±0.2	5.0±0.3	5.4±0.3	3.1±0.5	2.3±1
Co	0.2	0.2	0.2	<0.2	<0.2		1.5±1
Cu	<0.1	<0.1	<0.1	<0.5	<0.5		
S						34 ±4	

Sample 1-5 Incl. from Finland (Kouvo *et al.*, 1963).

Sample 6. Average of 16 determinations on mackinawite from the Mackinaw mine Evans *et al.*, 1964).

Sample 7. Mackinawite from the Muskox intrusion.

with pyrrhotite and pentlandite in the same section, using the same instrument.¹ The Vickers hardness of the pentlandite and pyrrhotite were found to average 210 and 286, respectively. These measurements fall well within the ranges published for these minerals (Bowie and Taylor, 1958) and impart confidence to the very low value found for the mackinawite.

The Muskox mackinawite shows a moderate bireflectance in both air and oil ranging from pink to pinkish grey. It is similar in color to pyrrhotite and may be slightly lighter or darker depending on its pleochroism position and degree of polish. Its anisotropism is strong, varying from greyish white/dark grey with nicols completely crossed, to bluish white/sienna brown with the upper nicol 1 or 2 degrees off the extinction position. These anisotropism colors are characteristic and, once recognized, were found helpful in determining the distribution of the mineral through the intrusion. It was noted that the intensity of the anisotropism is strongest after polishing and that it decreases markedly after a few weeks, though tarnish is not obvious. Repolishing restores the original

¹ Leitz Miniload Hardness Tester, using Vickers diamond with 50 gram load.

anisotropism. Most of the optical and physical properties of mackinawite are listed in Table 4 where they are compared with those observed for valleriite.

The x -ray powder-diffraction pattern of the Muskox material has been compared with patterns published for mackinawite from the Mackinaw mine and Outokumpu. The pattern for the Muskox iron sulfide originally

TABLE 3. X-RAY POWDER-DIFFRACTION DATA FOR MACKINAWITE

Least Squares ¹ Parameters		Muskox ²	
hkl	d (Å, calc.)	d (Å, obs.)	I
00 $\bar{1}$	5.035	5.03	S
101	2.967	2.96	M
110	2.597	2.60	VVW
002	2.518	—	—
111	2.308	2.31	VS
102	2.077	—	—
200	1.8365	1.84	M
112	1.8077	1.81	S
201	1.7253	1.73	M
003	1.6784	1.68	VW
211	1.5616	1.56	M
103	1.5265	1.52	W
202	1.4837	—	—
113	1.4096	1.41	W
212	1.3757	—	—
220	1.2986	1.29	M
004	1.2588	1.26	W
221	1.2574	—	—
203	1.2389	1.24	W
104	1.1908	—	—
301	1.1897	—	—
213	1.1739	—	—
310	1.1615	—	—
222	1.1511	—	—
114	1.1327	1.13	M
311	1.1318	—	—
302	1.1010	—	—
312	1.0547	1.05	S
204	1.0383	—	—
223	1.0271	1.03	MB
005	1.0070	—	—

¹ Data from Evans *et al.*, 1964.

² Muskox sample with 7 reflections of pentlandite omitted. FeK α radiation, camera diameter 57.3 mm. VS=very strong, S=strong, M=medium, W=weak, B=broad.

TABLE 4. COMPARISON OF OPTICAL AND PHYSICAL PROPERTIES OF MACKINAWITE AND VALLERIITE FROM THE MUSKOX INTRUSION

Mackinawite		Valleriite
Color	pinkish gray close to pyrrhotite	dull bronze similar to graphite
Bireflectance	moderate to high pink to gray	extreme under high power creamy bronze to purple
Anisotropism	high, but decreasing with time after polishing. under completely crossed nicols: grayish white to dark gray under incompletely crossed nicols: bluish white to Sienna brown (diagnostic)	extreme white to bronze, remaining much the same only less intense under incompletely crossed nicols.
Reflectivity	not measured, but in same range as pyrrhotite	not measured, but very low, in same range as graphite
Vickers hardness (50 g load)	58	30
Polish	fair, always with scratches	very poor, showing semi-matte surface despite all efforts to polish

contained seven pentlandite reflections. When these were deleted 17 reflections remained which match very closely the calculated patterns (Table 3) as well as those given for the earlier-described mackinawite.

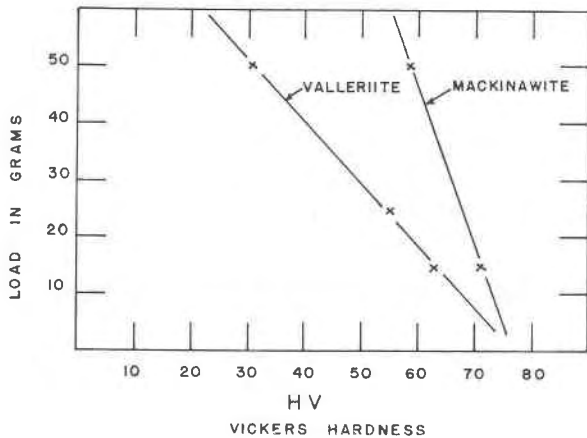
Valleriite. As stated, recent work by Evans *et al.* (1962, 1963) on the long-questioned composition of valleriite indicates a composition close to CuFeS_2 . It is apparent from this work that the problem of obtaining pure samples of the mineral is a formidable one even in cases where relatively large quantities are available. In the case of the Muskox valleriite, this problem is accentuated by the small quantities so far available, and it was decided not to attempt a chemical analysis at this time.

Some of the physical and optical properties of the Muskox valleriite are discussed as follows (see also Table 4). It was not found possible to polish the mineral beyond a semi-matte surface, even in small grains. This characteristic seems diagnostic for the Muskox valleriite when com-

paring it to relatively well-polished grains of mackinawite. The valleriite color is typically an overall dull bronze. Under higher powers in air or oil this resolves itself into a mosaic of platy pale bronze and purplish blue grains which expresses the extremely high birefractance of the mineral.

The Vickers hardness of valleriite varies between 30 and 63, showing a systematic increase with decreasing load on the diamond indenter (Table 5). With 50 gram loads, which probably result in more meaningful values than with the lighter loads, the Vickers hardness of valleriite is approximately half that of mackinawite.

TABLE 5. COMPARISON OF VICKERS HARDNESS OF MACKINAWITE AND VALLERIITE UNDER VARIOUS LOADS



The Muskox valleriite gives an x-ray powder diffraction pattern closely similar to the type material from Kaveltorp, Sweden as given by Evans *et al.* (1964). The characteristic pattern shown for the Muskox valleriite in Table 6 conclusively establishes its identity, and no attempt was made to deduce single-crystal patterns. The powder diffraction data given by Berry and Thompson (1962) for Kaveltorp valleriite also agree well with those found in the present study.

OBSERVED PHASE RELATIONS

Mackinawite in the Muskox intrusion is always found adjacent to, or in contact with, pentlandite and this is considered to be a significant association. Mackinawite most typically occurs as irregular masses within larger pentlandite grains. The outlines of the mackinawite grains may be somewhat flame-like or show a shape-dependance on the usually well-developed pentlandite cleavage (Figs. 2, 3). The maximum grain diam-

TABLE 6. X-RAY POWDER-DIFFRACTION DATA FOR VALLERIITE

Calculated ¹		Muskox Valleriite ²	
hkl	d (Å)	d (Å, obs.)	I
00.3	11.367	11.53	VS
00.6	5.683	5.74	VS
00.9	3.789	3.81	W
10.1	3.269	3.25	S
10.2	3.225	—	—
10.4	3.064	—	—
10.5	2.959	—	—
00.12	2.842	2.87	W
10.7	2.723	—	—
10.8	2.601	—	—
—	—	2.51	W
10.10	2.365	2.37	W
00.15	2.273	2.29	W
10.11	2.254	—	—
10.13	2.050	2.06	M
10.14	1.956	—	—
11.0	1.896	—	—
00.18	1.894	1.89	M
11.3	1.870	1.865	—
11.6	1.799	1.80	M
10.16	1.788	—	—
10.17	1.712	—	—
11.9	1.696	—	—
20.1	1.640	1.635	VW
20.2	1.634	—	—
00.21	1.624	—	—
20.4	1.612	—	—
20.5	1.596	—	—
11.12	1.577	—	—
10.19	1.575	—	—
20.7	1.556	—	—
20.8	1.532	1.54	W

¹ Data from Evans *et al.*, 1964.

² Muskox valleriite sample: pattern run on Fe K α radiation, camera diameter 57.3 mm. VS=very strong, S=strong, M=medium, W=weak.

eter observed for mackinawite is approximately 0.2 mm. while the average is perhaps one tenth of this figure. Mackinawite may take up to 50 per cent of the area of a given pentlandite grain.

Where pentlandite and mackinawite are the only sulfides present, a third phase, native nickel-iron of composition in the awaruite range generally rims the pentlandite. In such cases, the awaruite is commonly

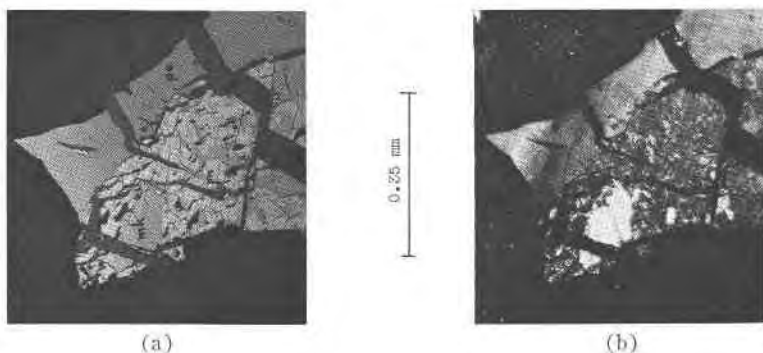


FIG. 2A. Mackinawite (mk) intimately associated with pentlandite (pn). Pyrrhotite (po) extends along right-hand side. Black cross-cutting veinlets are magnetite. Serpentine matrix. (Sample E2180') Incident illumination, oil immersion.

FIG. 2B. Same field as Plate 2A. Note high anisotropism of mackinawite. Incident illumination as in 2-A but with fully crossed nicols.

but by no means invariably separated from the pentlandite-mackinawite assemblage by an intermediate phase (magnetite) or by a narrow void.

Mackinawite has also been observed in contact with cubanite, chalcopyrite, valleriite and serpentine. The latter two of these contact relationships show little consistency, appearing for the most part to be sporadic and coincidental, and are relatively rare. Pyrrhotite is present in most sulfide assemblages, but shows little tendency to be directly associated

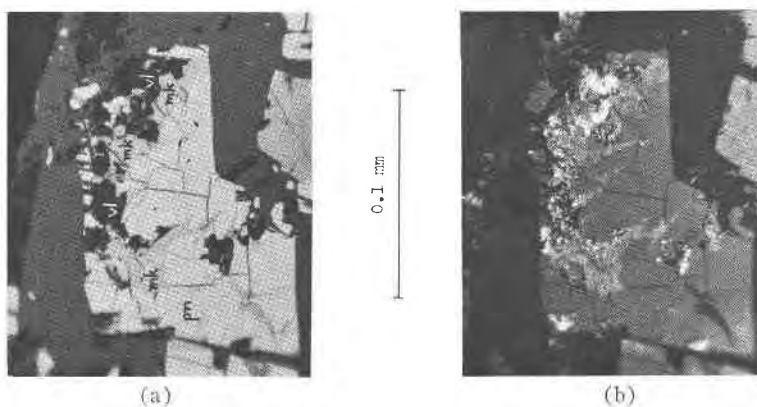


FIG. 3A. Mackinawite (mk) associated with valleriite (vl) in pentlandite (pn) (Sample N3305.8'). Incident illumination, oil immersion.

FIG. 3B. Same field as Plate 3A. Note high anisotropism of mackinawite and extreme anisotropism of valleriite. Incident illumination as in 3A but with fully crossed nicols.

with mackinawite. Chalcopyrite is rather uncommon in the central and lower parts of the layered series and no significant relationship to the mackinawite seems indicated. Cubanite was observed in only two of the mackinawite-bearing sections examined. Like mackinawite, it is associated with pentlandite where it occurs as irregular branchworks, or as sub-parallel, flame-like lamellae. In this association, the textural relationships of both cubanite and mackinawite to pentlandite appear to be much the same. In the two cases where cubanite and mackinawite were present in the same sections, they looked to be in contact, but their mutual relationships were not clear.

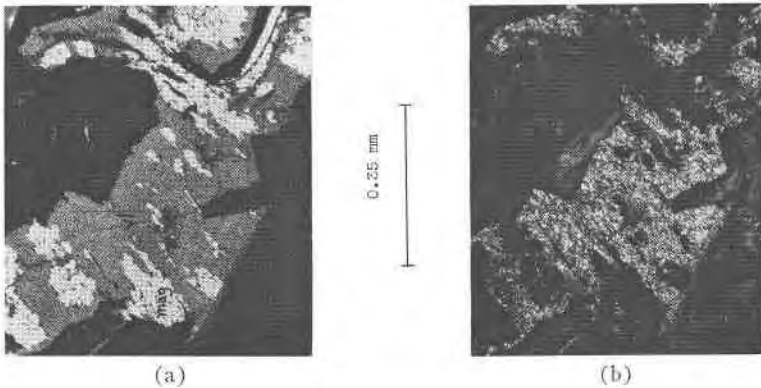


FIG. 4A. Valleriite (vl) replacing magnetite (mag) veinlet in serpentine (Sample N3305.8'). Incident illumination, oil immersion.

FIG. 4B. Incident illumination as in 4A but with fully crossed nicols.

Other minerals present in mackinawite-bearing rocks include native copper, chalcocite, native iron and a cobalt-iron phase with an approximate composition $\text{Co}_{50}\text{Fe}_{50}$. These phases occur in the same general parts of the layered series as mackinawite but have not been observed in contact with it. A description of the mineralogy and occurrence of these unique metallic phases in the Muskox intrusion is the substance of a separate paper presently in preparation.

Valleriite occurs as a minor constituent of serpentine where it may be alone, or intimately associated with secondary magnetite formed during the serpentinization process (Figs. 4A,B). It also occurs in contact with mackinawite and pentlandite, as stated, but this seems to be an exceptional assemblage (Figs. 3A,B). The valleriite generally occurs as sinuous threads or bands which, like much of the magnetite, conform to the cleavage structure of enclosing serpentine. It shows little tendency to be directly associated with other sulfides, including mackinawite.

DISTRIBUTION

Several hundred polished sections of surface samples and approximately 140 polished sections of core samples from the MuskoX deep drilling program were examined during present and related studies. Mackinawite was not recognized in any of the surface samples but was identified in 37 samples of drill core from the central layered series and in two samples from the marginal zone. Except for its absence in the sulfide-deficient core zone (Fig. 1), the mackinawite-pentlandite assemblage occurs intermittently downward from the chromite horizon to the base of the layered series, and into the inner part of the marginal zone. The rocks in this part of the intrusion consist of dunites, peridotites and olivine-bearing pyroxenites, all of which are serpentinized to some degree. Above the chromite horizon, the rocks become dominantly gabbroic rather than ultramafic, and both pentlandite and mackinawite are rare or absent in sulfide assemblages. In the marginal zone, where the rocks grade inward from bronzite gabbro at the contact through picrite to peridotite, the sulfide assemblages commonly include pentlandite, but virtually no mackinawite except in the partly serpentinized peridotites. One sample from the South hole near the main intrusive contact contains mackinawite in a pentlandite veinlet in gabbro, but this appears to be epigenetic. Mackinawite has not so far been observed in the MuskoX feeder or in any of the diabase dikes of the area.

Valleriite was observed in about a dozen core specimens from widely differing locations in the three deep drill holes, but is probably of more general occurrence than this would suggest. It occurs below the chromite horizon within what now may be termed the "mackinawite zone" and the suggestion is that the overall pattern of distribution of valleriite may be similar to that of mackinawite.

ORIGIN

The following remarks are of a preliminary nature and are offered prior to any published description of the MuskoX sulfides except as outlined by Smith (1962). They are put forward to indicate the present direction of thinking of the senior author, and revisions may be necessary as work proceeds. The present discussion is limited in scope by the fact that both mackinawite and valleriite are associated, at least in time, with several unusual phases as mentioned in the preceding section. No theory can properly explain the formation of individual phases in a system without considering the entire assemblage involved. This will be more appropriately attempted in a subsequent paper discussing the

occurrence and distribution of sulfides and related minerals through the entire intrusion.

Textural evidence consistently indicates that mackinawite has formed by the selective replacement of pentlandite, or possibly some earlier sulfide associated with pentlandite, such as cubanite. Among the factors suggesting that mackinawite did not exsolve from pentlandite during cooling are: (1) mackinawite occurs as irregular grains in pentlandite, never as oriented intergrowths, and (2) the shape of the mackinawite grains is partially controlled by the prominent pentlandite cleavage. More indirect evidence further suggests that the mackinawite formed at moderate or low temperatures during early stages of, and as a result of serpentinization of olivine. In general support of this is the fact that the three documented occurrences of mackinawite¹ are closely associated with serpentine. Also, secondary magnetite formed in Muskox dunites during the serpentinization process is closely associated in space with mackinawite. The mackinawite generally appears to have formed earlier than the magnetite, as suggested by the more or less typical textures shown in Fig. 2A.

The two earlier descriptions of mackinawite suggest that the mineral formed, at least in part, by secondary processes. In the Mackinaw mine, some of the mackinawite, which is referred to as valleriite in the original description (Milton and Milton, 1958), is believed to be a replacement of pentlandite. Other occurrences of mackinawite in chalcopyrite from the same deposit are said to be (p. 438) "probably formed by exsolution although the textures could have been formed by replacement as well." The mackinawite from the Outokumpu mine (Kouvo, *et al.*, 1959, 1963) is described as being mainly a replacement of pentlandite. Moreover, it is interesting to note that the mineral was found in the same deposit occupying open tension cracks in earlier-formed sulfides such as chalcopyrite, pyrrhotite and sphalerite.

No temperatures of formation have been suggested for natural mackinawite, but it seems significant that Berner (1962) was able to synthesize the mineral in a strongly reducing environment from aqueous solution at room temperature and atmospheric pressure. He also discovered mackinawite in the sediments of the Mystic River in Boston where it formed as a corrosion product of iron scrap in the presence of H₂S.

As stated, mackinawite has not been identified in samples collected from surface exposures of the Muskox intrusion. Other examples of differences in sulfide assemblages between surface samples and those

¹ Mackinaw mine, Washington (Milton and Milton 1958); Outokumpu mine (Kouvo, *et al.* 1959, 1963) Finland; Muskox intrusion.

taken at depth from stratigraphically equivalent units have been noted. These include the presence of marcasite in pyrrhotite in most surface and near-surface samples, and its almost complete absence at depth. In the case of mackinawite, its absence in surface samples is not surprising since, according to Berner (1962, p. 669) wetted mackinawite "oxidizes rapidly in air to FeOOH (lepidocrocite) and orthorhombic sulphur."

Little information exists on the occurrence of valleriite. Older descriptions are frequently unreliable because the possibility exists that the mineral referred to as valleriite may in fact be mackinawite. Fortunately, however, the textural evidence on the origin of the mineral in the Muskox intrusion itself appears to be interpretable with some confidence. The occurrence of valleriite in serpentine-rich rocks as sinuous bands parallel to the cleavage calls for a replacement origin after or during late stages of serpentinization. Valleriite commonly replaces magnetite which formed as a result of the serpentinization process (Fig. 4A) and this lends further support to the proposition that the valleriite formed late and at low temperatures.

In summary, then, the mineralogical and theoretical evidence as outlined indicate that both mackinawite and valleriite formed as replacement minerals at submagmatic temperatures. The mackinawite formed as an alteration in pentlandite, probably during intermediate stages of serpentinization, while the valleriite replaced serpentine and secondary magnetite after or during later stages of the same event. Observations made to date indicate that conditions favorable to the formation of mackinawite were met only in parts of the central and lower layered series and in the inner parts of the marginal zone. The essential requirements seem to be the presence of olivine and pentlandite as early phases followed by reducing conditions accompanying serpentinization. The instability of mackinawite in normal weathering environments is confirmed by its absence in pentlandite-bearing surface rocks which are stratigraphically equivalent to those containing the mackinawite-pentlandite assemblage at depth.

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