ALTERATION OF PHLOGOPITE TO CORRENSITE AT SHARBOT LAKE, ONTARIO[1]

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Abstract—A low-charge corrensite, i.e., a regular 1:1 interstratified smectite/chlorite was found in veins and fissures of an extensively fractured dolomite south of Perth, Ontario, Canada. Total chemical analysis indicated that the mineral was trioctahedral, having a structural formula corresponding to:

$$(Ca_{0.17})(Mg_{7.36}Al_{0.90}Fe^{3+}_{0.45}Fe^{2+}_{0.42})^{VI}(Si_{6.06}Al_{1.94})^{IV}O_{20}(OH)_{10}.$$
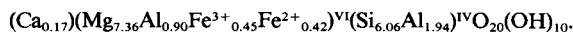
The coefficient of variation (CV) of the d(00l) spacings calculated for the mineral from X-ray powder diffraction data was 0.42, well below the maximum recommended value for corrensite, 0.75. The corrensite coexists with phlogopite, which is present as small (~0.5 mm) crystals throughout the rock and locally as large (~5 cm) crystals in pegmatitic veins. Microscopic observations of large phlogopite crystals partially altered to corrensite suggest that corrensite formed at the expense of phlogopite by hydrothermal alteration.

Key Words—Corrensite, Hydrothermal alteration, Interstratification, Phlogopite.

Résumé—Une corrensite, minéral interstratifié régulier 1:1 smectite/chlorite, a été trouvée dans les veines et les fissures d'un affleurement dolomitique très fortement fragmenté au sud de Perth, Ontario, Canada. L'analyse chimique totale a montré que le minéral était trioctaédrique avec une formule structurale correspondant à:

$$(Ca_{0.17})(Mg_{7.36}Al_{0.90}Fe^{3+}_{0.45}Fe^{2+}_{0.42})^{VI}(Si_{6.06}Al_{1.94})^{IV}O_{20}(OH)_{10}.$$
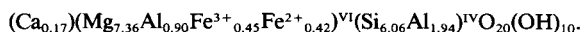
Le coefficient de variation (CV) des espacements d(00l) calculé pour le minéral à partir des données de RX était de 0,42, soit bien en deça de la limite recommandée, 0,75. La corrensite coexiste avec la phlogopite qui est présente sous forme de petits cristaux (~0,5 mm) dans tout l'affleurement et aussi sous forme de grands cristaux (~5 mm) dans des veines de pegmatite. Un examen microscopique de plusieurs grands cristaux de phlogopite partiellement altérés en corrensite a suggéré que celle-ci est formée aux dépens de la phlogopite par altération hydrothermale.

INTRODUCTION

Regular 1:1 chlorite/swelling mineral interstratification (corrensite) has been known for many years (Lippmann, 1956; Earley *et al.,* 1956; Bradley and Weaver, 1956). Such minerals may be of hydrothermal (Alietti, 1957; Sudo and Shumoda, 1978; April, 1980; Brigatti and Poppi, 1984) or sedimentary (Earley *et al.,* 1956; Bradley and Weaver, 1956; Johnson, 1964; Kübler, 1973; April, 1981; Proust, 1982) origin. Recently, Velde (1985) reviewed the occurrences of corrensite and concluded that it forms in a variety of geologic environments, somewhat in opposition to the conclusion reached by Millot (1964) who considered corren-site to be a product of supersaturation conditions. A regular interstratified chlorite/swelling mineral has also been produced synthetically by the oxidation of chlorite (Ross and Kodama, 1976).

Corrensites are predominantly trioctahedral, although dioctahedral analogues also exist (Brown *et al.,* 1974; Nishiyama *et al.,* 1975). The Clay Minerals Society Committee on Nomenclature (Bailey *et al.,* 1982) recommended that the name corrensite be given to a 1:1 regular interstratification of trioctahedral chlorite and either trioctahedral smectite or trioctahedral vermiculite. To define corrensite better, use of the adjectives "low-charge" and "high-charge" was further recommended by the Committee for chlorite/smectite and chlorite/vermiculite, respectively.

Recently, corrensite was found in eastern Ontario in a metamorphosed dolomite that also contained phlogopite. Whereas reports in the literature frequently mention an association of corrensite and chlorite, no such association of corrensite and phlogopite has been reported. The objective of this study was therefore to

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investigate the possible genetic relation between phlogopite and corrensite and to provide further information on the mineralization processes in marbles of eastern Ontario, for which the presence of interstratified or expanding minerals has not been documented.

MATERIALS AND METHODS

Location and geology

The site is located along highway 38 near Saint-George Lake, 44°43′N and 76°40′W, 35 km southwest of Perth, Ontario. This area of Precambrian Grenville rocks consists chiefly of diorite, gabbro, gneiss, dolomite, and marble (Jupe and Jackson, 1964). The marbles belong to the Hadrynian group (1310 M.Y. old, Silver and Lumbers, 1965) and have been mapped as metasedimentary rocks (Wynne-Edwards, 1957). They occur <8 km from the Frontenac axis; metamorphic conditions were severe enough to generate granulite facies rocks (Bourne, 1974). Coarse-grain skarns are present and are most common in areas of extensive metamorphism where the marbles are found (Shaw et al., 1963).

The chief metamorphic processes which affected the rocks of the Grenville were regional metamorphism and metasomatism (Hewitt, 1956). Contact metamorphism has been effective locally (Hewitt, 1956). In particular, three consecutive stages of metamorphism have been recognized (Wynne-Edwards, 1967) in the study area: (1) plastic deformation of all metamorphic rocks; (2) plastic flow of crystalline limestone against more rigid gneissic units; and (3) local cataclasis and faulting.

At the site, dolomite is exposed in a roadcut 135 m long and as much as 6 m high. The outcrop is extensively fractured. Phlogopite is common in this dolomite and occurs as millimeter-size crystals and as blocks as wide as 6 cm in diameter throughout the rock. It amounts to about 5% of the rock and about 90% of many veins and fracture fillings. Black graphite flakes, about 0.2 mm in diameter, are common. In some dolomite samples, traces of talc, hematite, and ankerite were identified by X-ray powder diffraction. Corrensite occurs as a greenish clay, crumbly, alteration product in veins 10–60 cm across. It also occurs in pockets. About 40 individual occurrences of corrensite were noted in this roadcut. Bulk material was collected at several places in the outcrop for laboratory analysis.

Separation and analytical methods

Coarse fragments were removed by hand from the samples. The <5-mm material was then dispersed using ultrasonic vibration, and the <50-μm fraction was collected by sedimentation and centrifugation, with subsequent separation of the <2-μm fraction. Four distinct corrensite specimens (samples 1–4) were treated in this manner and characterized; however, because of
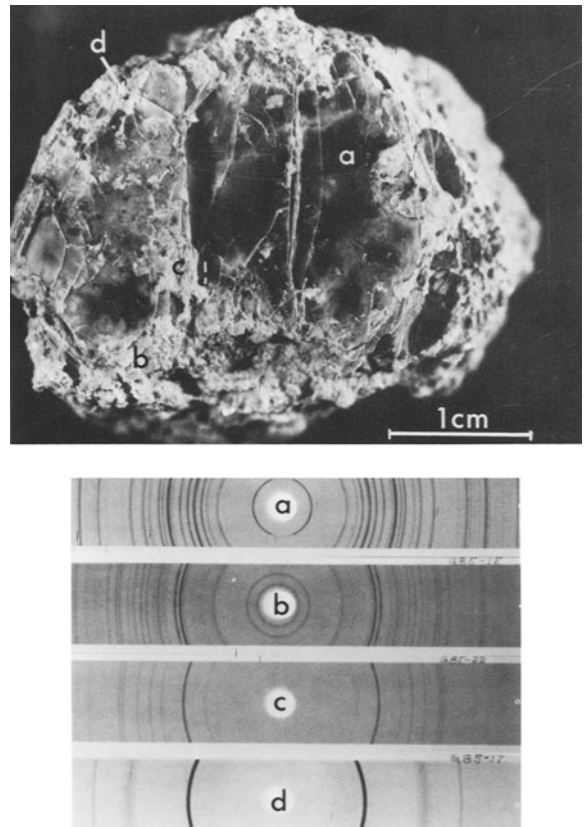

Figure 1. Mineral assemblage observed in a phlogopite crystal book and XRD patterns recorded with a Gandolfi camera: a, phlogopite; b, corrensite; c, calcite; d, graphite.

sample similarities, the data for sample 2 only will be reported.

Samples were examined by X-ray powder diffraction (XRD) after saturation with Mg, Ca, or K before and after glycerol solvation using a glycerol-ethanol mixture (Miles and De Kimpe, 1985) or after heat treatment at 550° and 700°C. Analyses were carried out on an automated Scintag Pad V (CoKα radiation, 40 kV, 40 mA, graphite monochromator) diffractometer for powder samples or using a Gandolfi camera for untreated grains. Total chemical analysis was performed by atomic absorption after fusion with $Sr(BO_2)_2$ and dissolution with HNO_3. Differential thermal (DTA) and thermogravimetric (TGA) analyses using a Fisher analyzer and a Cahn electrobalance, respectively, and infrared (IR) analysis by the KBr-pellet method using a Beckman IR 4250 spectrophotometer were also used to characterize the corrensite samples. Optical microscopy and scanning electron microscopy (SEM), the latter coupled with energy-dispersive X-ray (EDX) analysis capability on a Cambridge Mark II Stereoscan instrument, were used to examine the mineral assemblages.
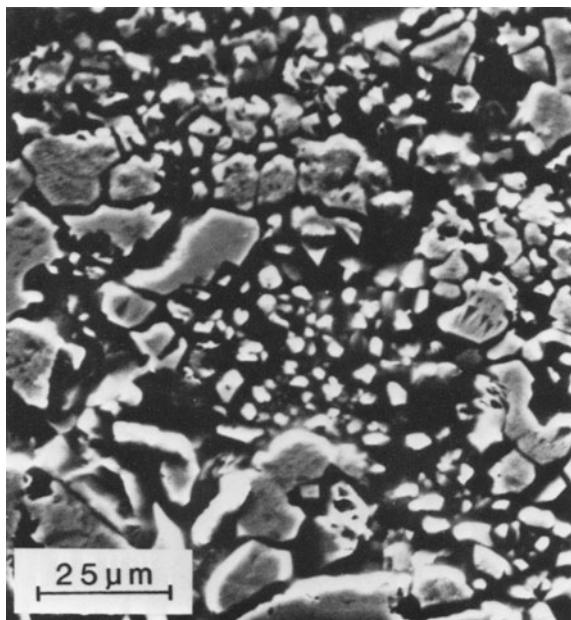
Figure 2. Photomicrograph of etched figures at the surface of calcite crystals.
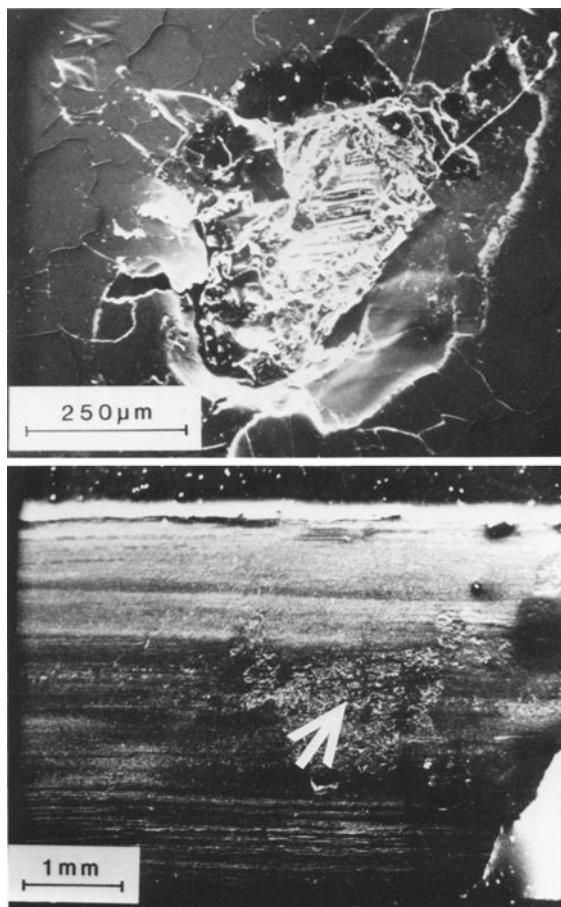

Figure 3. Scanning electron micrographs of surface (upper) and cross section (lower) of a phlogopite book, showing the alteration to corrensite (indicated by the arrow).

RESULTS AND DISCUSSION

Microscopic observations

Phlogopite books, 3–5 cm in diameter and 1–2 cm thick, collected from a vein in the dolomite outcrop, were examined by optical microscopy (Figure 1). The normal amber color of phlogopite was faded in various degrees giving rise to irregular surface patterns. The bulk of each book consisted of phlogopite, but many cavities and depressions of various sizes and shapes were present on the surface, filled with greenish-white powdery material. The latter was also more abundant at the edges of books and commonly penetrated into cleavages. A carbonate mineral, which appeared to be calcite, was common between the phlogopite flakes. Under the microscope it bubbled intensely immediately upon addition of one drop of dilute HCl. Etched figures on the calcite crystals (Figure 2) indicated an alteration, possibly by a hydrothermal solution. Many black specks, 0.1–0.5 mm in size, were also present. Different mineral grains were removed from the mica books with a needle and tweezers and identified by XRD (Figure 1) as phlogopite, corrensite, calcite, and graphite.

Surface and cross-section microscopic study of a phlogopite book indicated a possible genetic relation between phlogopite and the corrensite. Surface examination of a cavity (Figure 3, upper) showed that some layers were altered from the surface inwards and replaced by corrensite, whereas a cross-section examination of the cavity area (Figure 3, lower) revealed an apparent transformation of phlogopite at depth. EDX analysis of the cross section (Figure 4) showed a considerable decrease in K, a moderate decrease in Si, almost no change in Mg, a moderate increase in Fe, and a large increase in Al in the cavity area with respect to the relatively unaltered phlogopite.

X-ray powder diffraction analysis

XRD of a glycerol-solvated, Mg-saturated sample of corrensite showed a long spacing having an integral series of higher orders (Figure 5, curve 1). The XRD patterns of specimen 2, before and after cation saturation, glycerol solvation, and heat treatment, were used to characterize the component layers. The Mg-exchanged sample gave a first-order d(001) value of about 28 Å (curve 2), which expanded to about 32 Å upon glycerol solvation (curve 3), suggesting that the expandable component was smectite. The first-order spacing shifted to 24 Å after the sample was heated to 550°C (curve 4) and remained at this value upon heating at 700°C (curve 5). The 24-Å spacing corresponds
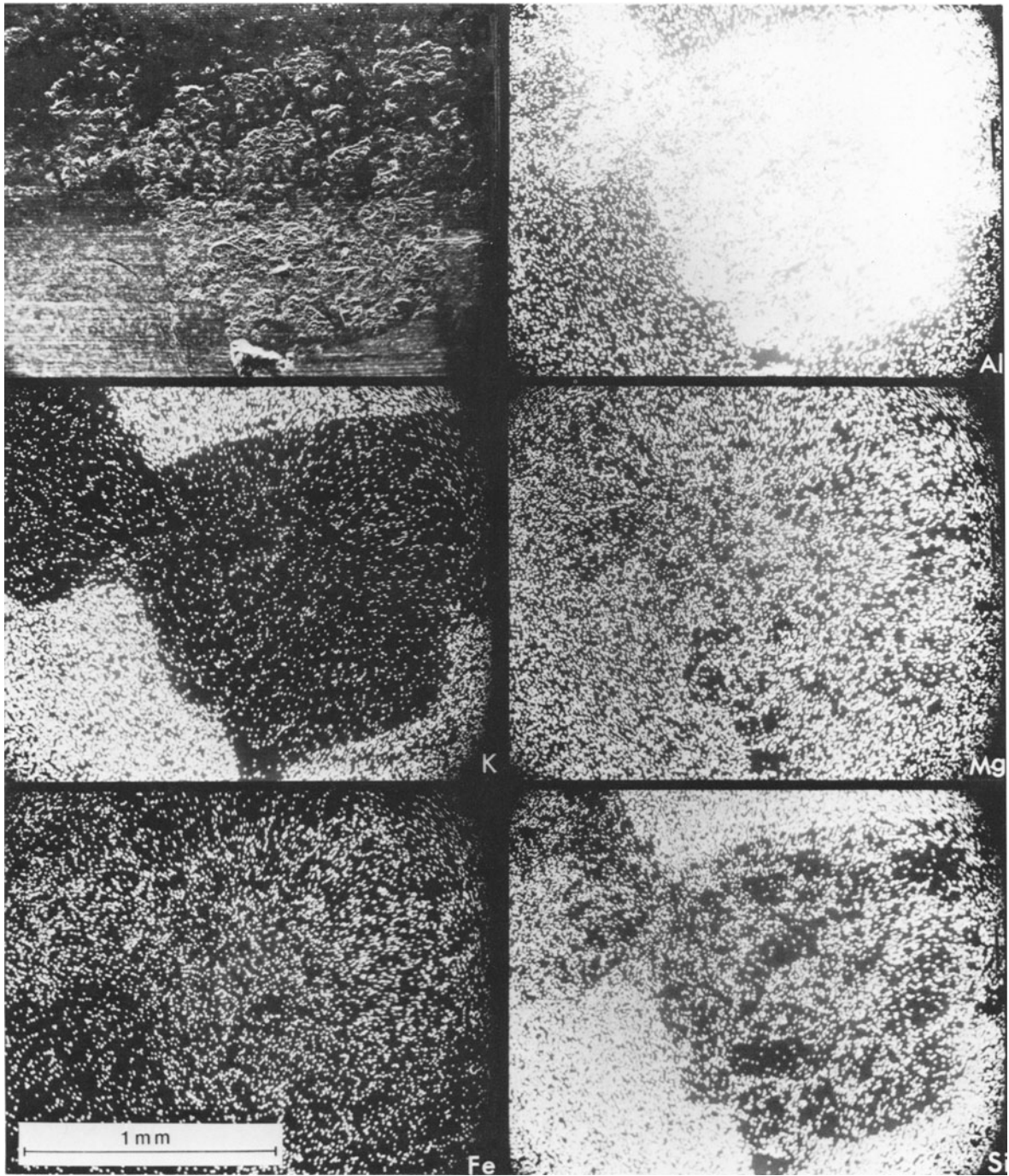
Figure 4. Microprobe analysis of the cross section shown in Figure 3, presenting the cation distribution in the altered section. Top left micrograph shows the cross section.

to the sum of a 14-Å component (chlorite layer) and a 10-Å component (collapsed smectite layer). The first-order spacing of the K-saturated sample was 28 Å (curve 6); it expanded to about 32 Å after heating at 300°C and glycerol solvation (curve 7), thereby ruling out the presence of a vermiculite component in this interstratified mineral (MacEwan, 1961). The absence of a vermiculite component was also supported by the expansion of the sample to 32 Å after glycerol solvation and Ca saturation (curve 8).
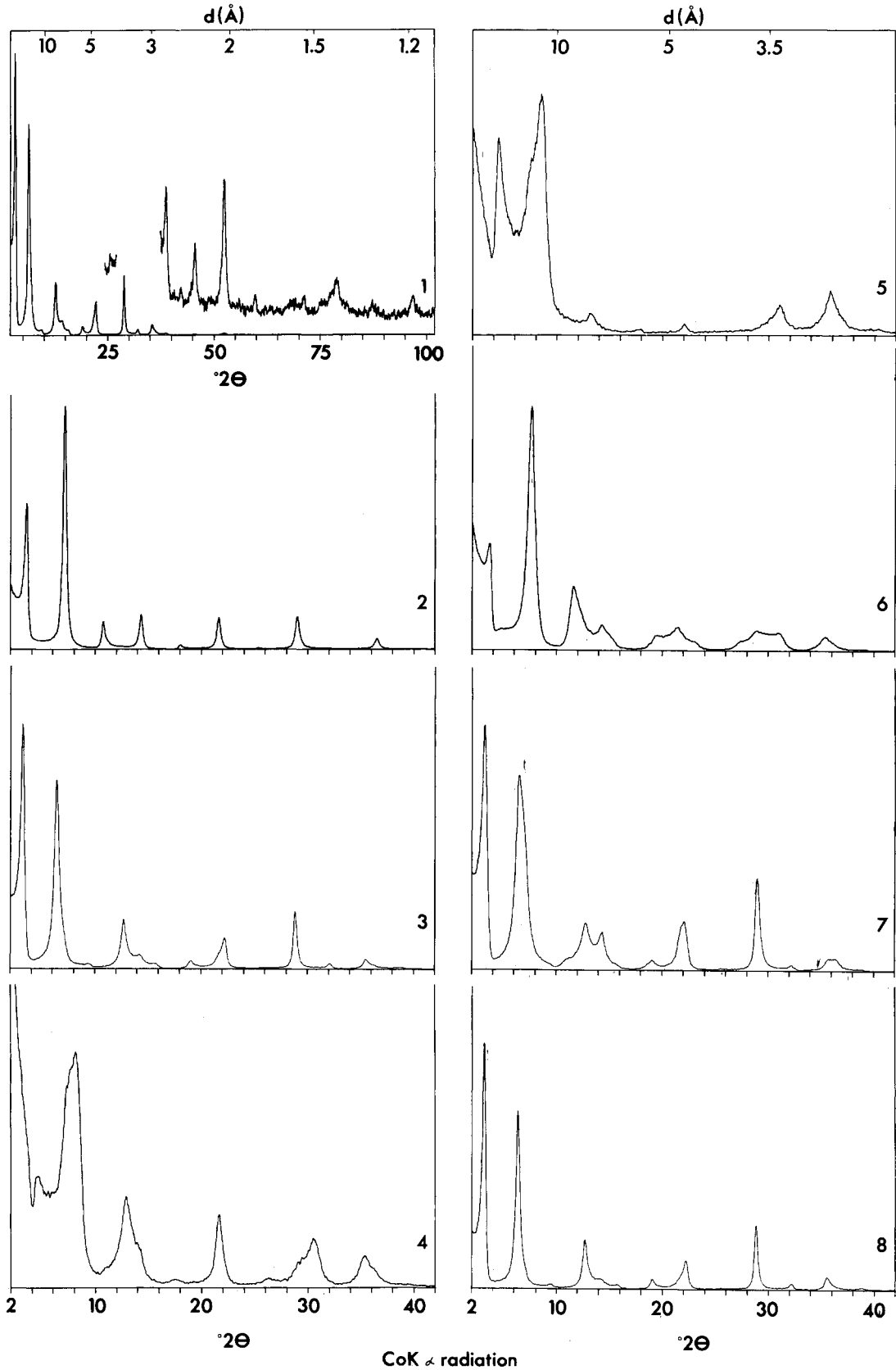
Table 1.   X-ray powder diffraction 00*l* data for a glycerol-solvated, Mg-saturated corrensite sample.

l	d (Å)	Calculated d(001) (Å)	I	Relative I
1	31.60[1]	31.60	3773	100
2	15.82	31.65	2810	74.5
3	10.70	32.10	24	0.6
4	7.986	31.94	641	17.0
5	6.446	32.23	29	0.8
6	5.332	31.99	137	3.6
7	4.589	32.12	311	8.2
8	4.002	32.01	7	0.2
9	3.561	32.04	902	23.9
10	3.206	32.06	42	1.1
11	2.906	31.96	207	5.5
12	2.675	32.10	13	0.3
13	—	—	—	—
14	2.288	32.03	12	0.3
15	2.134	32.01	5.4	0.1
16	2.005	32.08	19.5	0.5
17	—	—	—	—
18	1.783	32.09	4.3	0.1
19	1.688	32.07	1.4	0.04
20	1.598	31.96	5	0.1
21	1.527	32.06	2.2	0.06
22	1.454	31.98	5	0.2
23	1.396	32.10	5.7	0.15
24	—	—	—	—
25	1.285	32.12	2.2	0.06
26	1.231	32.00	1.4	0.04
27	1.190	32.13	6.5	0.2
28	1.146	32.08	1.4	0.04
29	—	—	—	—
30	1.068	32.04	1.4	0.04

Average d(001) = 32.02 Å, s = 0.135, CV = 100 s/average d(001) = 0.422.

[1] Numbers given in italics are of low accuracy.

The mineral therefore appears to consist of two components—chlorite and smectite. Nadeau *et al.* (1984) suggested that interstratified minerals are composed of aggregates of fundamental particles. The regularity of higher order spacings was therefore examined by the coefficient of variation (CV) test recommended by Bailey *et al.* (1982). The mean d(001) value calculated from 26 basal reflections of a glycerol-solvated, Mg-saturated sample (Table 1) was 32.02 Å. The CV for d(001) was 0.42, well below the recommended value, 0.75. Thus, the sample appears to be a 1:1 regularly interstratified chlorite/smectite. The 060 reflection of a non-oriented specimen was 1.535 Å, indicating the trioctahedral nature of the mineral (Bailey *et al.*, 1982), which is further supported by the chemical data discussed below. Therefore the mineral meets all qualifications of corrensite.
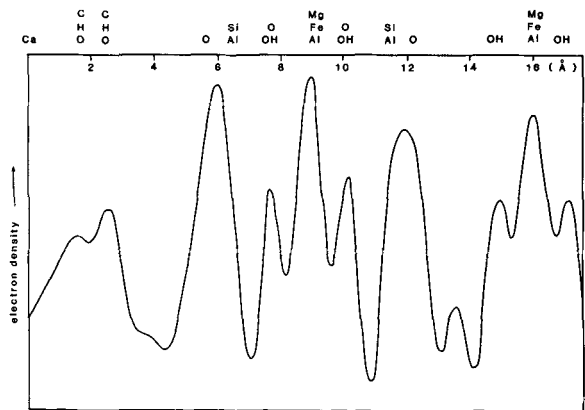

Figure 6.   One-dimensional Fourier synthesis of corrensite.

A one-dimensional Fourier synthesis was carried out using the $|F_{obs}|$ values (Table 1). The signs of the structure factors were calculated for a synthetic corrensite (Ross and Kodama, 1976) that was similar to the one presently studied. The Fourier synthesis (Figure 6) confirmed the alternate sequence of two types of component layers, chlorite and smectite.

Chemical analysis

Total chemical analyses were performed on the major minerals identified in this rock outcrop (Table 2). The chemical composition of dolomite gave a nearly theoretical formula. Phlogopite and corrensite compositions differed from each other mainly by the amount of Fe and H_2O+ and by the absence of K in corrensite, compared with phlogopite. Sesquioxides, extractable by dithionite-citrate-bicarbonate reagent mixture (Mehra and Jackson, 1960), amounted to 0.2% Fe_2O_3 and 0.1% Al_2O_3. Structural formulae were calculated (see Foster, 1960) from Table 3 data on the basis of 22 oxygen atoms for samples that were considered by XRD to be pure. Ti^{4+} was allocated to the octahedral layer (Deer *et al.*, 1962). For corrensite, the calculation was based on 50 negative charges $(O_{20}(OH)_{10})$, as proposed by Brigatti and Poppi (1984). Six-fold coordinated Al represented about 10% of the octahedral cations in samples 1 and 2 and about 14% in samples 3 and 4; $(Fe^{2+} + Fe^{3+})$ made up 8.9–9.8% of the octahedral layer. All corrensite samples were thus chemically similar. Whereas the chemical analysis of pure specimens of phlogopite and corrensite showed only small differences in the Al_2O_3 content (Table 3), the larger difference in the Al_2O_3 content as measured by

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Figure 5.   X-ray powder diffractograms of <2 μm corrensite samples prepared as oriented film on glass slides: (1) Mg-saturated and glycerol-solvated, showing enhanced weak reflections over the range 2°–102°2θ; (2) Mg-saturated; (3) Mg-saturated and glycerol-solvated; (4) Mg-saturated and heated at 550°C; (5) Mg-saturated and heated at 700°C; (6) K-saturated; (7) K-saturated and glycerol-solvated after heating at 300°C; (8) Ca-saturated and glycerol-solvated.

Table 2. Chemical analyses (wt. %) of the different minerals present in the dolomite.

Oxide	Dolomite		Phlogopite		Corrensite			
	1	2	1	2	1	2	3	4
SiO_2	0.00	0.00	39.2	39.6	36.3	35.2	36.3	38.8
Al_2O_3	0.70	0.30	17.5	14.4	13.1	14.0	15.0	12.9
Fe_2O_3	0.00	0.00	0.80	0.95	3.08	3.48	2.53	3.57
FeO	0.58	0.58	tr	tr	2.90	2.90	3.76	2.46
TiO_2	tr	tr	1.60	1.00	0.15	tr	0.15	0.10
P_2O_5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
MnO	0.18	0.07	0.01	0.01	tr	tr	tr	tr
CaO	35.5	32.5	1.10	2.00	0.90	0.93	0.80	1.00
MgO	17.5	21.5	27.0	28.0	28.5	28.5	26.0	26.0
K_2O	0.15	0.00	8.25	8.75	tr	tr	0.30	tr
Na_2O	0.00	0.00	1.10	0.55	tr	tr	tr	tr
H_2O+	n.d.	n.d.	2.09	3.73	11.6	10.6	11.2	11.0
H_2O-	0.09	0.07	0.16	0.11	3.58	4.28	3.02	4.20
CO_2	44.9	46.00	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Total	99.60	101.02	98.81	99.10	100.11	99.89	99.06	100.03

n.d. = not determined; tr = traces.

EDAX (Figure 4) suggests the presence of noncrystalline, Al-rich material in the phlogopite book cavity.

The cation-exchange capacity of sample 2, determined at pH 7 with 1 N NH_4OAc, was 36.6 meq/100 g of clay, with 85% of the exchange sites being occupied by Ca^{2+}.

Thermal analyses

The DTA curve (Figure 7) was similar to that obtained by Lippmann (1954) for corrensite, originally called a regularly interstratified "swelling chlorite"/chlorite mineral. The two endothermic peaks at 80° and 160°C shown in Figure 7 are typical of smectite in which exchange sites are saturated with divalent cations (Mackenzie, 1972). The major endotherm at 575°C

and the exotherm at 835°C are attributed to the chlorite component (Bradley and Weaver, 1956; Mackenzie, 1972).

The TGA curves (Figure 8A) of material from two particle size classes are similar and comparable to those reported for corrensites (Earley *et al.*, 1956; Brown *et al.*, 1974; Brigatti and Poppi, 1985). About 2% water was lost at <300°C, about 8% between 300° and 650°C, and about 3% at >650°C. The TGA differential curves (Figure 8B) showed the largest loss between 500° and 600°C.

Infrared (IR) absorption analysis

The IR curve (Figure 9) was similar to that reported for natural corrensite (Veniale and Van der Marel, 1970;

Table 3. Structural formulae of phlogopite and corrensite samples, based on 44 and 50 negative charges, respectively.

	Phlogopites		Corrensites			
	1	2	1	2	3	4
Octahedral atoms						
Mg	5.548	5.868	7.344	7.362	6.709	6.648
Al	0.191	—	0.883	0.897	1.282	1.201
Fe^{3+}	0.082	0.100	0.297	0.450	0.326	0.456
Fe^{2+}	—	—	0.415	0.416	0.539	0.350
Ti^{3+}	0.164	0.104	0.020	—	0.020	0.012
Mn	0.001	0.001	—	—	—	—
Tetrahedral atoms						
Si	5.370	5.533	6.236	6.062	6.245	6.614
Al	2.630	2.367	1.764	1.938	1.755	1.386
Interlayer atoms						
K	1.443	1.561	—	—	0.066	—
Ca	0.161	0.299	0.166	0.172	0.148	0.183
Na	0.291	0.149	—	—	—	—
Layer charge						
Octahedral	0.409	0.350	1.418	1.597	1.380	1.003
Tetrahedral	−2.630	−2.768	−1.764	−1.938	−1.755	−1.386
Interlayer	2.096	2.308	0.332	0.344	0.362	0.366
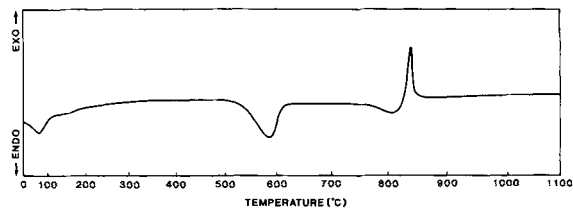
Figure 7.   Differential thermal analysis curve of corrensite.

Figure 9.   Infrared spectrum of natural corrensite, in pressed KBr pellet.

Bergaya *et al.,* 1985) and for the regularly interstratified chlorite/swelling chlorite mineral of Van der Marel and Beutelspacher (1976). Although the octahedral layer of the corrensite sample (Table 3) contained a high proportion of Mg atoms (7.35 atoms), the 660-cm^{-1} doublet was not clearly separated into the bands having maxima at 670 and 650 cm^{-1}, which was assigned by Farmer (1974) to the libration of Mg$_3$OH in trioctahedral 2:1 phyllosilicates and which was also reported by Bergaya *et al.* (1985) for a corrensite from Italy.

SUMMARY AND CONCLUSIONS

The analytical results reported above indicate that the greenish white mineral sampled near Saint-George Lake, Ontario, is corrensite. Formation by degradation of chlorite (Brigatti and Poppi, 1984) is not acceptable here, because no *discrete* chlorite was found. Corrensite was observed as powdery masses on and within phlogopite books, having ragged boundaries with the host mineral. Corrensite probably formed at the expense of phlogopite by the addition of Mg^{2+} ions between K-depleted layers. Formation of an increasing number of "chlorite" layers alternating with the smectite layers probably induced the corrensite crystallization. A similar accretion process was proposed by Grim *et al.* (1960) for the formation of corrensite at the expense of illite in an evaporite in New Mexico. A phlogopite-corrensite assemblage was not reported by Velde (1985) in
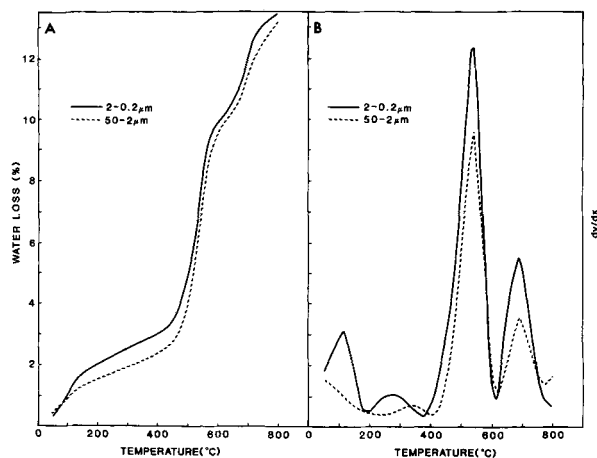
his review, and thus the formation of corrensite from phlogopite appears to be another mechanism of formation for this mineral.

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Figure 8.   Thermogravimetric (A) and differential thermogravimetric (B) curves for two size classes of particles.

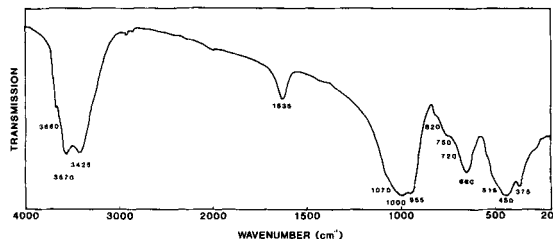Farmer, V. C. (1974) *The Infrared Spectra of Minerals:* Mineralogical Society, London, 539 pp.

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