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LAYER SILICATES FROM SERPENTINITE-PEGMATITE CONTACT (WIRY, LOWER SILESIA, POLAND)

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Abstract—Highly tectonized contact between serpentinite and younger pegmatite in the magnesite mine of Wiry contains various layer silicates. Vermiculite, chlorite, smectite, and interstratified mica-vermiculite were recognized by means of routine XRD examination. Two three component interstratifications of mica-vermiculite-chlorite and chlorite-swelling chlorite-smectite were identified by a combined procedure of deconvolution of the XRD patterns and simulation of XRD tracings. A mineral with large diffraction maxima, displaying "chlorite intergrade" characteristics, appeared to be a mixture of chlorite, mixed layer chlorite-smectite, and vermiculite. Polytypes of phyllosilicates were determined by the X-ray transmission method. Due to the heritage of parent mineral polytype structure by transitional products of alteration, two distinct sequences of layer silicates were observed: one formed from trioctahedral mica (vermiculite, mixed layer mica-vermiculite); and one evolved from chlorite (e.g., mixed layer chlorite-swelling chlorite-smectite). A tentative scheme of the primary contact zone structure, not obscured by subsequent brittle tectonics either by transformation of layer silicates, is proposed.

Key Words—Chlorite, Intergrade, Mica, SEM, Serpentinite/pegmatite contact, Three component interstratified minerals, Vermiculite, Weathering, X-ray powder.

INTRODUCTION

Occurrences of layer silicate assemblages in altered rocks are often linked with transformation processes responsible for the formation of transitional phases and secondary layer silicates, which develop due to gradual changes in the interlayer of a parent mineral (Nagasawa *et al.*, 1974; Noack and Colin, 1986; Proust *et al.*, 1986; Beaufort, 1987; de Kimpe *et al.*, 1987; Buurman *et al.*, 1988). Similar sequences of layer silicates were obtained during experimental alteration studies (e.g., Rich, 1968; Hoda and Hood, 1972; Ross and Kodama, 1974, 1976).

Layer silicates from contacts of granitic veins with serpentinite seem to be suitable material to check the reliability of the transformation sequences. Granite/serpentinite contact zones include at least three separate sectors: mica-rich, chlorite-rich, and talc-rich (Curtis and Brown, 1969). Each forms at the expense of the ultrabasic rock (Lvova and Dyakonov, 1973). The alteration products may be considered as formed from different parent minerals, or as formed due to direct precipitation from a solution.

EXPERIMENTAL MATERIALS AND METHODS

Geological setting

The magnesite mine of Wiry is located 50 km southwest of Wrocław (Lower Silesia, Poland) in the western part of the Jordanów-Gogołów serpentinite massif (about 350 Ma according to Pin *et al.*, 1989) close to the younger Strzegom-Sobótka Variscan granite massif (about 280 Ma, Pin *et al.*, 1989) (Figure 1). The magnesite is believed to be younger than the granite; however, its absolute age is unknown (Gajewski, 1970). Scarce slip surfaces suggest magnesite formation before the youngest brittle tectonics episode (Alpine?). Details on geology of the region are given by Majerowicz (1972, 1981) and Gajewski (1970).

The rocks from Wiry are characterized by their diversity and the apparently chaotic distribution of mineral assemblages (Figure 2). The main associations distinguished after careful examination of five contact zones are:

1) Serpentinites, usually with overgrowths of monoclinic colourless amphibole and dissected by a network of magnesite veinlets;
2) Pegmatite veins often dismembered by tectonic events into separate blocks;
3) Rocks rich in layer silicates (trioctahedral micas, chlorites, vermiculite, etc., exclusively of serpentines);
4) Veins of massive magnesite, partly dismembered as above;
5) Sheared veins of chlorite ± talc ± monoclinic amphibole cutting the serpentinite; and
6) A patchwork of veins, veinlets, and patches of sepiolite, smectite, and silica minerals.

Several episodes of brittle deformation dismembered an initial sequence of metasomatic zones formed between the serpentinite and the leucocratic rock in Wiry, creating a mosaic structure (Figure 2). The in-

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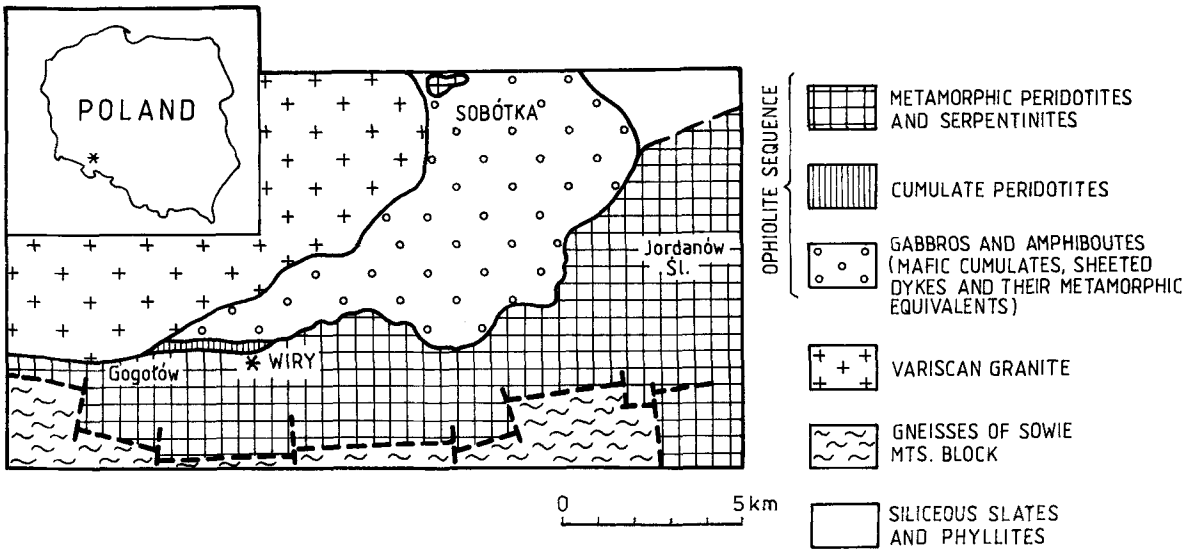


Figure 1. Simplified geological sketch map of the western and central part of Jordanów-Gogołów serpentinite massif, after Majerowicz (1981).

fluence of brittle tectonics was also recorded in thin sections of the rocks (Figure 3a). This paper focuses on the layer silicates that occur in the tectonized veins adjacent to the pegmatites (Group 3) and to veins rich in chlorite cutting the serpentinite (Group 5).

Samples

After macroscopic and thin section study and preliminary XRD examination of about 40 samples collected in the field, 11 samples were selected for the detailed study. Hand specimens are soft, friable, and usually fine-grained. However, well-pronounced schistosity, inherited from parent rock, is still easily distinguishable. Samples usually contain an admixture of flakes up to 2 mm in diameter (micas, black or grey; vermiculite, brown; chlorite, green; and talc, silver to white) as well as white or grey amphibole-rich fragments. Pegmatites are highly fractured, with schist fragments filling the fissures. Pockets of apatite prisms up to 1 cm in diameter, magnesite spots, and veinlets are commonly found.

Analytical methods

SEM. SEM observations were made with a Jeol-JSM-840A electron microscope equipped with an energy dispersive X-ray detector allowing qualitative chemical analysis. Fresh samples as well as specimens after ultrasonic treatment were used in the study.

XRD. The routine identification of layer silicates was performed on oriented specimens using a DRON-2A diffractometer and CoKα radiation. Samples were saturated with Na^+, K^+, and Mg^{2+}-ions, heated and treated with ethylene glycol and glycerol (both liquid). Some oriented preparations were heated on a homemade thermal stage, and their X-ray tracings were recorded at a temperature of 200°C using the Sigma 2070 reflection diffractometer made by CGR. These tracings, as well as transmission ones, were collected using the DRX program (a simple program for collecting and evaluation of X-ray diffraction data, Vila *et al.*, 1988).

Deconvolution of the overlapped diffraction bands was performed by means of the NICEFIT program assuming Gaussian peak profiles. Méring's (1949) diagrams modified by Drits and Sakharow (1976) were used to estimate component concentrations in the interstratified structures, and Reynolds' (1985) NEWMOD 2 program was used to simulate XRD diagrams for the estimated layer ratios of interstratified phases.

RESULTS

SEM study

The SEM observations enabled two groups of samples to be distinguished:

1) Samples containing large and rather fresh flakes of chlorite that often resisted disaggregation by the ultrasonic treatment (Figure 4A). Taking into account results of the XRD and qualitative EDX analysis, these flakes were tentatively identified as Mg-rich trioctahedral mica (flakes rich in K, Mg, Al), Mg-vermiculite, and/or Mg-chlorite (flakes rich in Mg and Al),
2) Samples consisting of large flakes almost completely covered by spongy coatings (Figure 4B). Ultrasonically cleaned samples contained fresh K-free flakes (Figure 5, vermiculite); other flakes with blistered surfaces, rich in Mg and K (Figure 5, trioctahedral micas); and relics of spongy coatings in

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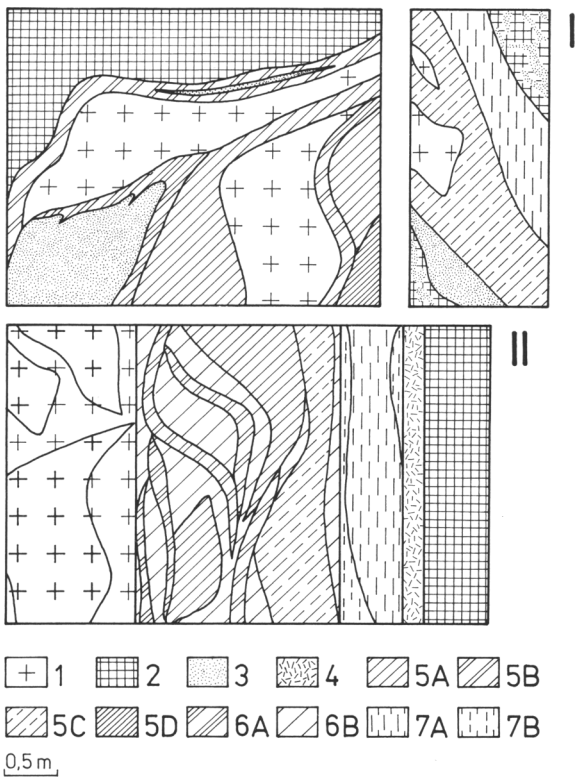


Figure 2. Geological sketch of pegmatite/serpentinite exemplary contacts in mine of Wiry: I = natural occurrence (schematic drawings of the mine side walls); II = reconstruction of the primary contact zone structure. Shapes of real samples (I) were fitted into the hypothetical zones from II (without brittle tectonic influence). 1 = pegmatite; 2 = serpentinite; 3 = magnesite; 4 = talc zone (hypothetical); 5 = rocks rich in vermiculite produced due to the trioctahedral mica alteration (±chlorite, ±talc, ±interstratified mica-vermiculite, ±mica, ±smectite, ±monoclinic amphibole, ±magnesite); 5A = sample W4; 5B = samples W3 (left on I) and W1 (right on I); 5C = sample W7; 5D = sample W6. In the mica zone, 6A = real sample W2 and 6B = hypothetical. In the chlorite zone, 7A = real sample W9 and 7B = hypothetical.

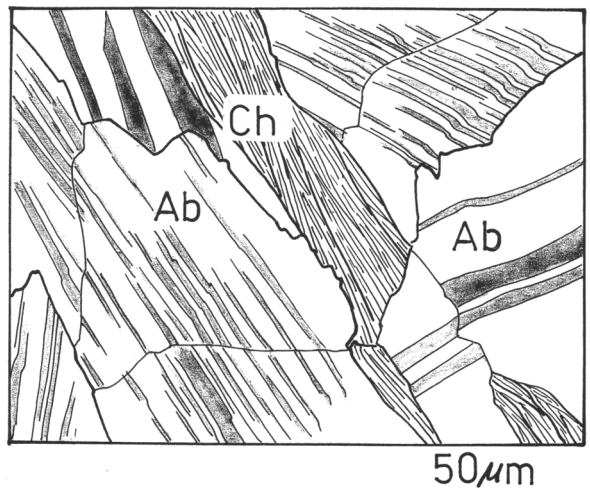


Figure 3. Highly tectonized albite (Ab) pegmatite, with chlorite (Ch) contact schist displaced into the pegmatite fracture, sketch after thin section, sample Wi106 (contact zone not shown in Figure 2).

fissures (Figure 4C and smectite spectrum on Figure 5) with a chemical composition similar to those in the fresh samples (Figure 4C).

Magnesite veinlets and coatings consisting of small rhombohedral crystals (Figure 4D) were ubiquitous. Veins of almond-shaped calcite were also present. The carbonates seem to have formed after the large flakes.

X-ray characteristics

The layer silicates, mica, smectite, vermiculite, chlorite, talc, and serpentine with minor admixtures of a monoclinic amphibole, feldspars, magnesite, quartz, and calcite were identified. Some samples rich in vermiculite (the vermiculite was identified according to criteria established by Brindley, 1966, and de la Calle and Suquet, 1988) contained quasi-regularly interstratified mica-vermiculite. The mixed-layer mineral did not expand after Mg^{2+} and liquid glycerol treatment; whereas, after heating of the K^+-sample at 500°C, the contracted 10 Å structure was produced (Figure 6). The computed X-ray pattern (Figure 7), agreed with the visual estimation (using Méring's 1949 diagram) of 40% mica layers and 60% vermiculite layers (Figure 5).

Figure 6 shows X-ray tracings of a mineral that can be tentatively identified as quasi-regularly interstratified mica-vermiculite. Cv of the ethylene glycol treated sample equal to 1.69 is too high for regular interstratification (as defined by Bailey, 1982). The mineral examined at 200°C in the heating stage mounted on the goniometer did not appear to be fully contracted (Figure 8). Peak positions were obtained by deconvolution of large experimental diffraction maxima (Figures 9a and 9b). The incomplete contraction during heating, as well as irrational series of the diffraction maxima, proved that the mixed-layer mineral contains interstratified, non-contracted 14 Å-layers. The content of 14 Å-layers was estimated at 25% using Méring's (1949) diagram modified by Drits and Sakharov (1976). The same volume for 14 Å layers was obtained by means of the method described by Jonas and Brown (1959). Thus, the interstratified mineral during heating still contained about 25% of 14 Å layers, which were undoubtedly chlorite.

Calculated diagrams similar to the experimental X-ray tracings of the mineral were prepared. The best agreement was obtained using the chemical composition of vermiculite from similar geological occurrence in Jordanów (within the Jordanów-Gogołów serpen-

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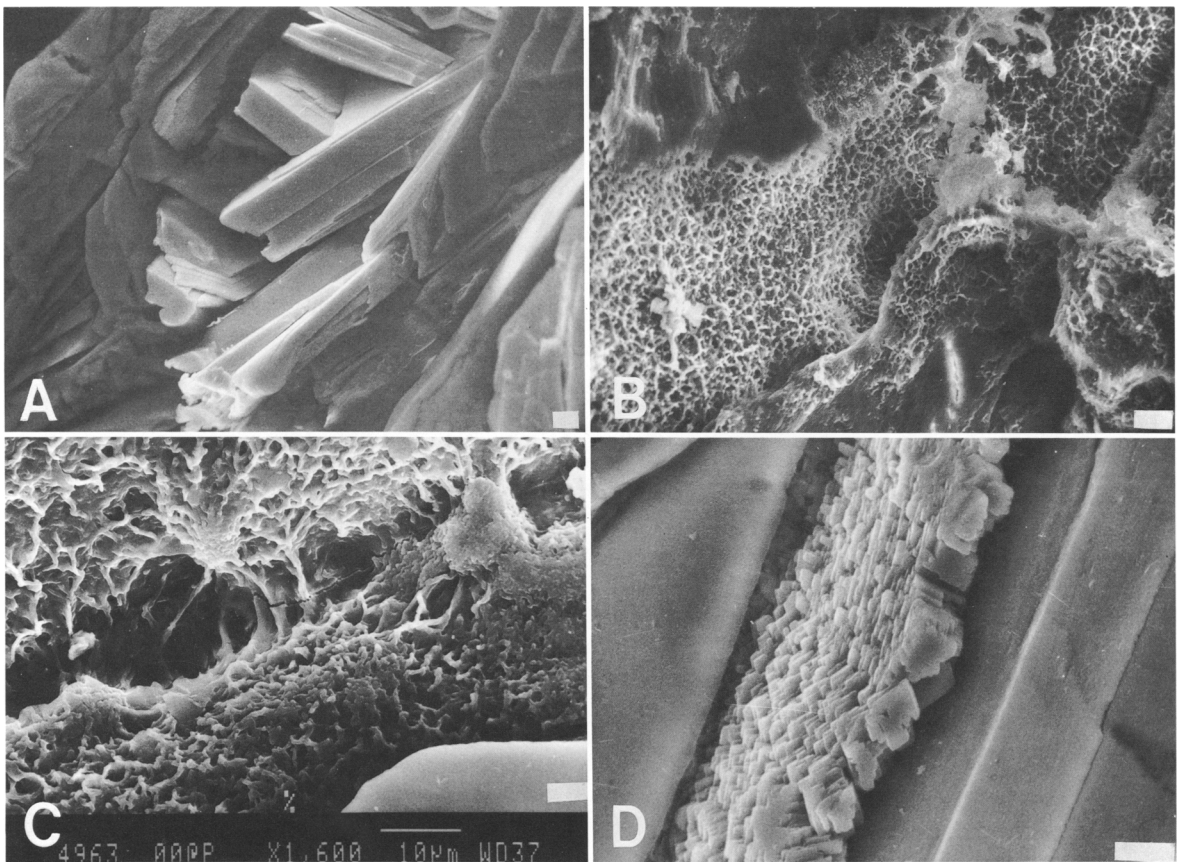


Figure 4. A) Fresh flakes of chlorite in sample W8 after ultrasonic treatment (UST), SEM; B) spongy coatings of a smectite covering large flakes of vermiculite, SEM, sample W3; C) blistering mica surface (1), large and fresh flake of vermiculite (2) and relic spongy aggregates of smectite (3), sample W1 after UST, SEM; D) rhomboidal small magnesite crystal in between large mica flakes, sample Wi5D after UST, SEM. Bar on SEM photographs represents 5 μm.

tinite massif) according to (Dubińska and Szafranek, 1990):

$$(Mg_{2.41}Fe_{0.41}Al_{0.37}Ni_{0.02}Cr_{0.02}Ti_{0.01})(Al_{1.05}Si_{2.95})O_{10}(OH)_2$$

and 40/60 vermiculite/mica ratio (Figure 10). The layer ratio in the interstratification was deduced to be:

1)	ethylene glycol		
		14Å—40%	(vermiculite + chlorite)
		10Å—60%	(mica)
2)	heating	14Å—25%	(chlorite)
		10Å—75%	(mica + vermiculite)

These data suggest that the interstratified mineral contains 25% 14 Å layers resistant to contraction (chlorite layers), 60% 10 Å layers of mica, and 15% layers that preserve a 14 Å structure after glycol saturation and contract during heating (vermiculite layers).

The procedure described above was applied to another three-component interstratified mineral (Figure 11). The K-saturated mineral (reflections 13.4 Å, 7.76 Å, 4.48 Å, and 3.53 Å) displayed characteristics of irregularly interstratified 14 Å layers (75%) and 10 Å layers (25%). After liquid ethylene glycol treatment (16.39 Å, 7.65 Å, 4.93 Å, 3.53 Å, and 2.82 Å), it contained about 40% 14 Å layers and about 60% expanded layers. Thus. the contents of layers of the interstratified mineral are:

1)	ethylene glycol		
		14Å—40%	(chlorite)
		10Å—60%	(smectite, swelling chlorite)
2)	heating		
		14Å—25%	(chlorite, swelling chlorite)
		10Å—75%	(smectite)

Vermiculite layers were eliminated from consideration to avoid any confusion. The above interpretation is tentative because of the likelihood of incomplete saturation of the mineral with K^+ ions and of a contraction of flakes at their edges that may have prevented the expulsion of water molecules from the interlayer.

An intergradient mineral was identified as a major

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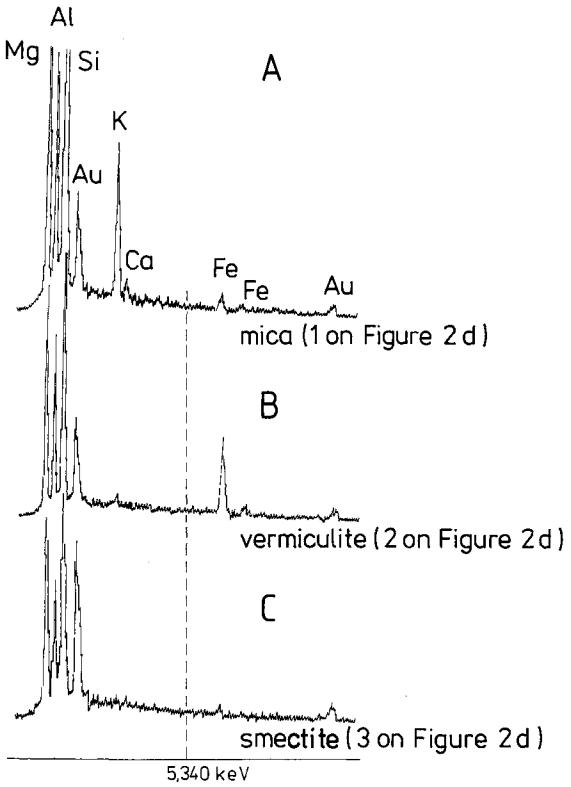


Figure 5. Energy dispersive X-ray spectra: A) blistering surfaces of mica, B) fresh flakes of vermiculite, C) relic smectite coatings in fissure (sample W1 after UST). Au = covering medium.

component of sample W9 (Figure 12). It did not completely contract after heating of K^+-saturated preparation. XRD patterns of the natural and Mg^{2+}-saturated preparations displayed vermiculite-like characteristics,

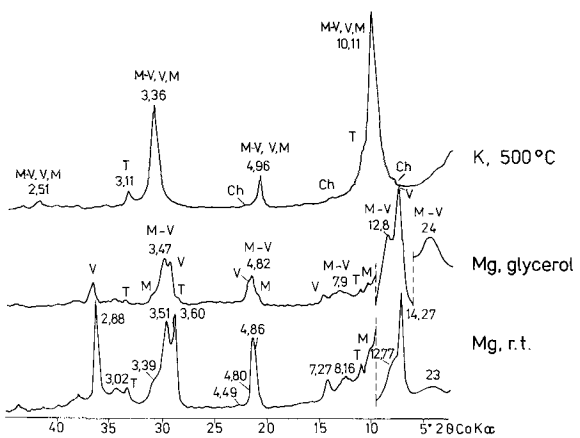


Figure 6. X-ray reflection tracings of sample W4 (4-16 μm): Ch = chlorite, M = mica, M-V = interstratified mica-vermiculite, T = talc, V = vermiculite, r.t. = room temperature. d-values were determined by deconvolution of experimental pattern. Some of them are almost hidden at first glance; their real position often differs from maxima at the tracings.

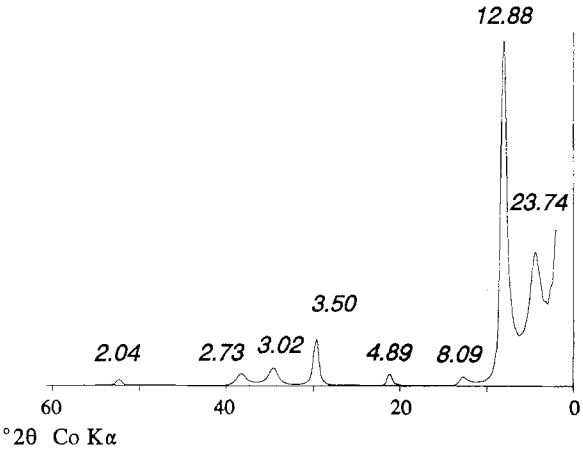


Figure 7. Calculated diffractometer pattern (CoKα) of interstratified trioctahedral (two water layer) Mg vermiculite (0.6)-trioctahedral mica (0.4) symmetrical distribution of iron. R = 1; d-values correspond to those on 6 (the lower pattern).

but the relative intensities of basal reflections were not typical for vermiculite (e.g., intensity of 005 reflection was too low). Saturation with ethylene glycol produced tailings of asymmetrical X-ray reflections. It is tentatively identified as a mixture of more than two layer silicates. The general status of intergradient minerals

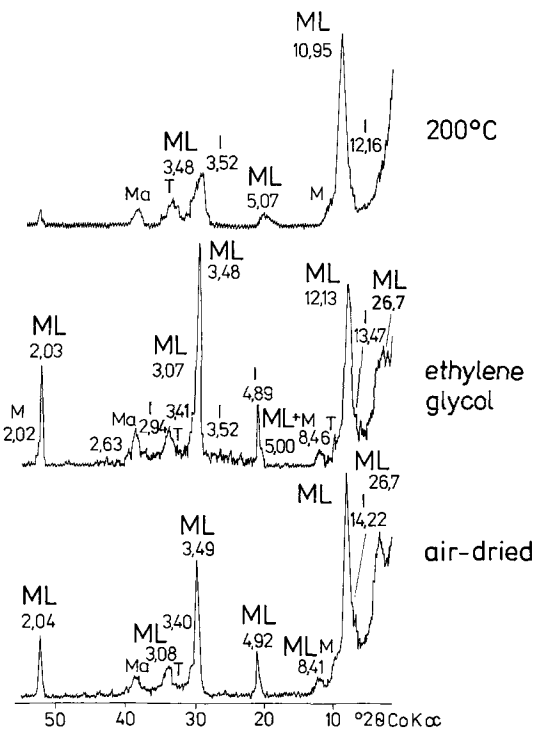


Figure 8. X-ray diffractograms of sample 3-W, reflection method: I = intergrade, ML = mixed layer. For other explanation, see Figure 6.

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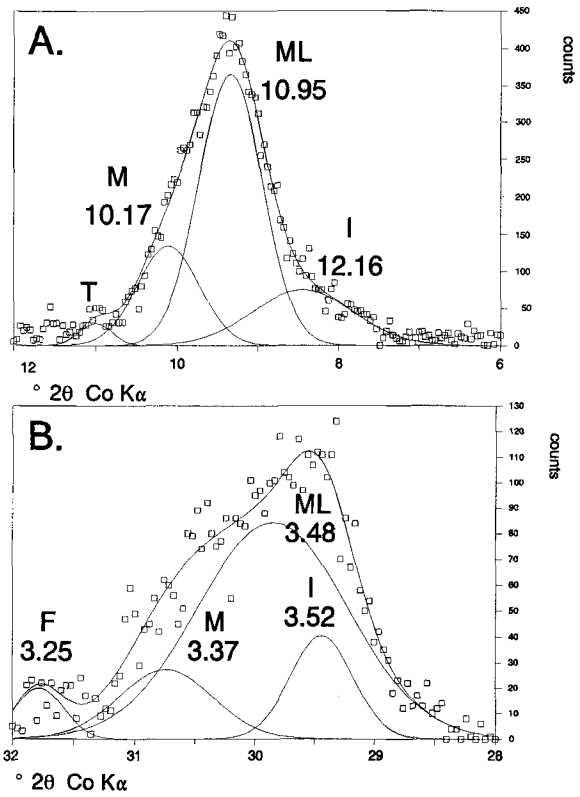


Figure 9. Deconvolution of large diffraction maxima (Figure 6, pattern of sample heated at 200°C in the ranges): A) 7°–11° 2θ, B) 28°–31.5° 2θ. Squares = experimental points, F = K-feldspar, I = intergrade, M = mica, ML = mixed layer, T = talc.

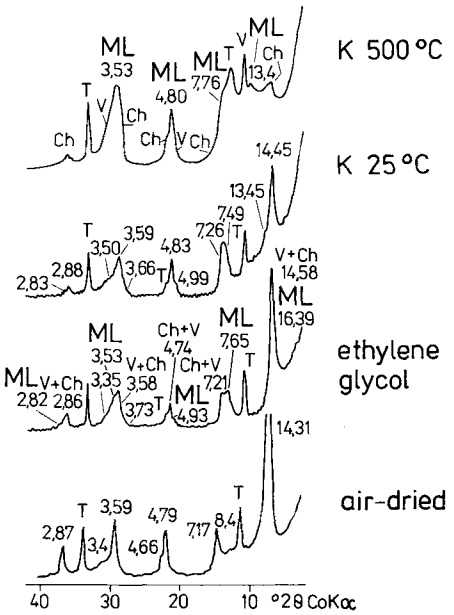


Figure 11. X-ray reflection tracings of sample W7 (ϕ < 0.5 μm), oriented aggregates, positions of overlapped peaks were determined by deconvolution of experimental pattern (some of them are almost completely hidden at first glance). ML = tri-component interstratified mineral chlorite (0.4)-swelling chlorite (0.35)-smectite (0.25). For explanation see text.

and their relation to mixed-layer chlorite-smectite, chlorite-vermiculite, and swelling chlorite is still obscure (Bailey, 1975; Barnhisel, 1977; Reynolds, 1988).

The large X-ray diffraction maxima ascribed to the intergrade structure were deconvoluted by means of the NICEFIT program. X-ray patterns of the K-saturated and heated as well as ethylene glycol treated samples, were used for the fitting. The pattern can be interpreted as due to the mixture of chlorite, vermiculite, and irregularly interstratified chlorite (55%)/smectite (45%) (Figure 12).

The samples with spongy coatings observed in SEM (Figure 4B) contain significant amounts of a smectite in the fine-grained fractions. Moreover, the samples

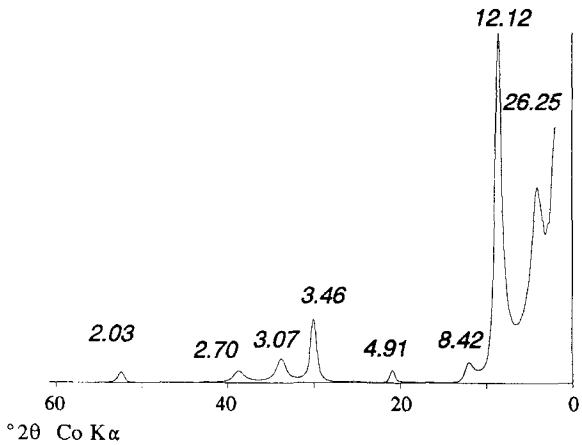


Figure 10. Calculated diffractometer pattern of interstratified trioctahedral mica (0.6)-trioctahedral Mg vermiculite (0.4). R = 1.

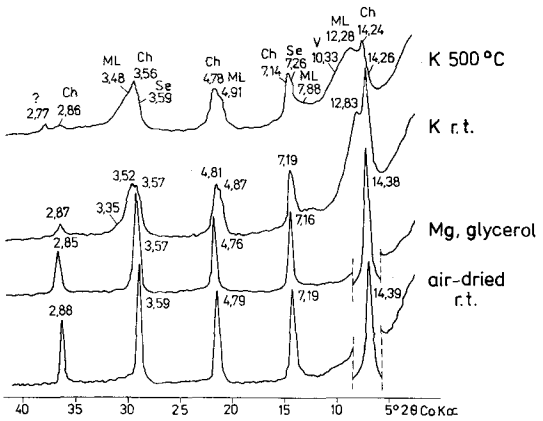


Figure 12. X-ray reflection tracings of sample W9 (ϕ < 0.5 μm), oriented aggregates. ML = irregularly interstratified chlorite (0.55)-smectite (0.45); Se = serpentine.

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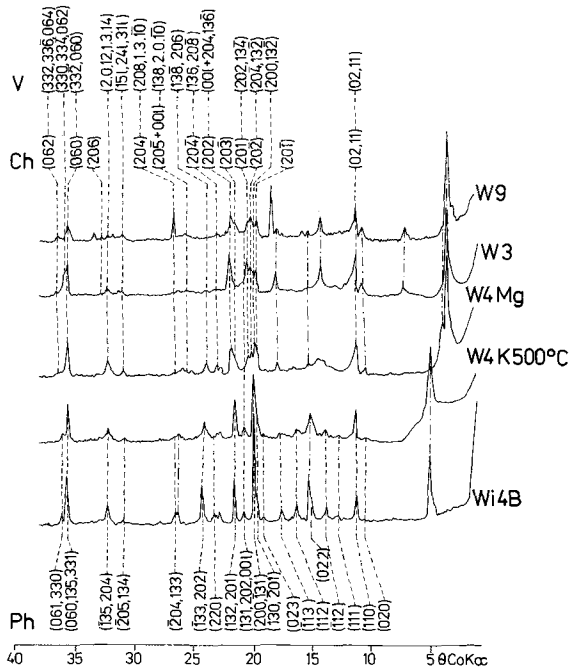


Figure 13. X-ray transmission patterns of the studied samples; $\nu = 90°$, V = vermiculite $2M_B$-VII-meso, Ph = phlogopite-like structure [$1M_A$-I ($3T_A$-IV)] produced after heating of K^+ saturated sample (e.g., W4 K 500°C) or natural phlogopite from Wiry (Wi4B); Ch = chlorite (subfamily C); indices after Bailey (1980); unspecified maxima represent (001) reflections.

initially rich in smectite became almost smectite-free after ultrasonic treatment. These observations indicate that smectite was the dominant component of the coatings. The smectite was also found in mica-rich samples that did not contain vermiculite.

The following polytypes were identified by means of the oblique texture X-ray method (Wiewióra and Weiss, 1985; Weiss and Wiewióra, 1986): 1) mica-$1M_A$-I ($3T_A$-IV), 2) vermiculite $2M_B$-VII-meso (Ia according to Bailey's (1980) notation, and 3) chlorite and some "intergradient" chlorite-vermiculite-subfamily C (IIb after Bailey, 1980). The K^+-saturated vermiculites and interstratified minerals containing mica layers resemble phlogopite-like structure [$1M_A$-I ($3T_A$-IV)] (Figure 13).

REMARKS ON THE ORIGIN

Sequence of layer silicate formation

Experimental studies of trioctahedral micas suggest that polytype analysis can be applied to determine the parent structure of micas completely altered and evolved into vermiculite (de la Calle *et al.*, 1976). The polytype analysis was also applied to determine the parent mineral in altered serpentinite-granite contacts in Szklary and Jordanów in Lower Silesia, where only highly evolved products of trioctahedral mica alteration were found (Wiewióra and Dubińska, 1987; Dubińska and Wiewióra, 1988). Minerals from Wiry (mica, mixed-layer mica-vermiculite, etc.) seem to represent an earlier stage of alteration and are more typical for low-temperature alteration (e.g., Lvova and Dyakonov, 1973) of trioctahedral micas.

Three-component interstratified minerals, vermiculite, and "intergradient" chlorite-vermiculite have preserved the structural characteristics of their parent minerals, i.e., vermiculite and mixed-layer minerals with mica layers readily regenerated phlogopite structures; whereas, "intergradient" minerals preserved chlorite structure. Samples rich in these altered minerals usually display well developed schistosity; hence, the major phyllosilicate evolution proceeded by transformation rather than by precipitation of newly formed minerals. A summary of the transformation of mica and/or chlorite in the Wiry area is shown in the schematic diagram of Figure 14.

Quasi-regularly interstratified mica-vermiculite was formed directly from phlogopite. Later chlorization of some vermiculitic layers produced a mica/vermiculite/chlorite mixed-layer mineral. Direct formation of chlorite from mica layer might also be possible (Nagasawa *et al.*, 1974; De Kimpe *et al.*, 1987).

The origin of the three component smectite/chlorite/swelling chlorite seems to be more complicated. There are at least two alternative possible ways of its formation:

1) Transformation of chlorite resulted in the development of mixed-layer chlorite/smectite, which is the substantial component of "intergradient" samples. Exchange of interlayer cations of smectite for hydroxide pillows produced the swelling chlorite layers.
2) The mineral evolved from vermiculite. The swelling chlorite layer can be attributed to a transitional stage of chloritization.

Both pathways for layer silicate transformation were observed in experimental alteration (e.g., Rich, 1968; Ross, 1975; de la Calle *et al.*, 1976) as well as in natural soils and altered rocks (Shimoda, 1970; Brindley and Souza, 1975; Proust *et al.*,, 1986; Inoue, 1987; Buurman *et al.*, 1988; Inoue and Utada, 1991).

The magnesite at Wiry precipitated from solutions of meteoric origin containing biogenic carbon (Jędrysek and Hałas, 1990). The same solutions are presumably responsible for formation of the smectite at Wiry.

Variation of the mineral assemblages in Wiry is probably a result of local equilibrium conditions as well as variations in solution composition during prolonged Tertiary weathering (by analogy with other similar occurrences in Lower Silesia region, e.g., Niśkiewicz, 1967). They were probably preceded by an older hydrothermal activity.

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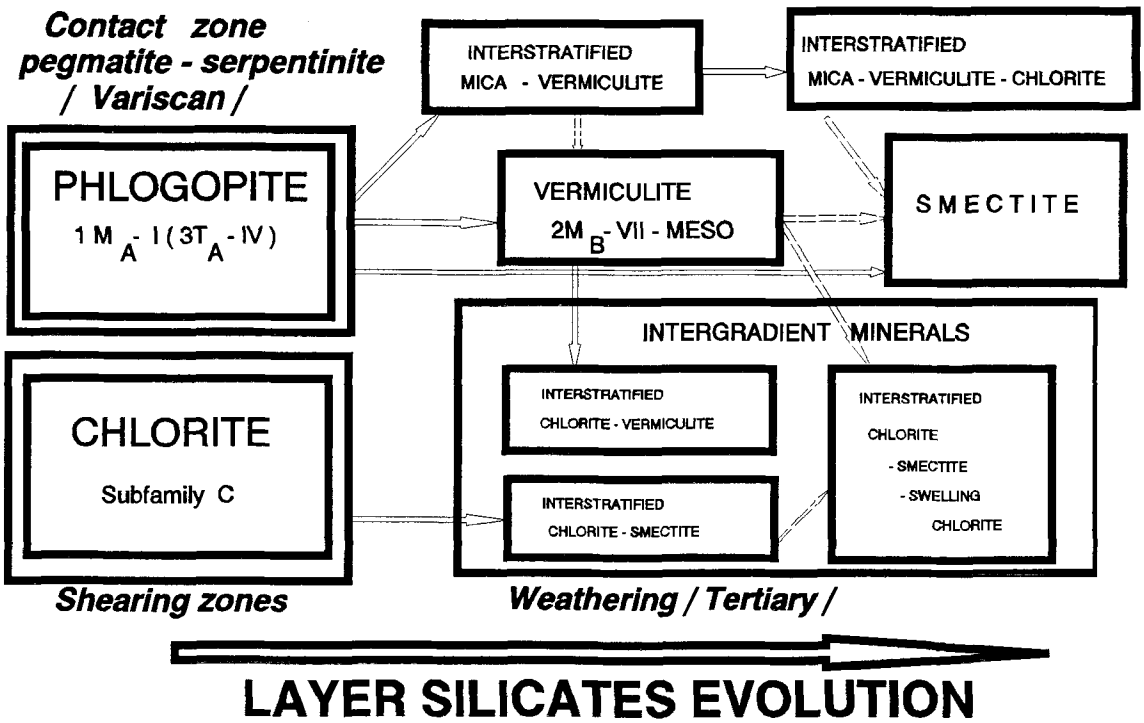


Figure 14. Schematic diagram of the layer silicates evolution sequences in the Wiry mine.

Evolution of the contact zone

The assemblage of layer silicates hitherto described evolved from two main parent minerals, mica and chlorite (both trioctahedral). Talc was also found; however, it has persisted unaltered to the present. Thus, the set of layer silicates is typical for low temperature contact zones between leucocratic rock (e.g., granite) and serpentinite, irrespective of their relative ages (Basset, 1963; Curtis and Brown, 1969; Sanford, 1982).

Results of field studies (typical zonation) were used for reconstruction of the initial contact zone structure in Wiry, i.e., without distortion due to brittle tectonics (Figure 1) and clearly aided in the interpretation of the alteration sequence. If the distortion had been ignored, the different mineral assemblages could have been improperly explained, e.g., as products of local equilibrium variations.

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