MICROMORPHOLOGY OF WEATHERING BIOTITE AND ITS SECONDARY PRODUCTS

Contribution no. 3 of the "Advisory Panel on Weathering Phenomena and Neoforma- tions" of the "Sub-Commission on Soil Micromorphology of the I.S.S.S."

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1. INTRODUCTION

1.1. GENERAL

This paper on the micromorphological aspects of biotite weathering is second in a series on the weathering of individual minerals. The series was started by Delvigne et al. (1979) with an article on weathering olivine. Papers on the weathering of muscovite and chlorite are in preparation.

The paper is mainly concerned with the transformations taking place in biotite during its weathering as observed in thin sections with the light microscope. To explain these transformations, however, reference has to be made to data from X-ray diffraction (XRD), wet chemical analyses and submicroscopic techniques.

Altered biotite lamellae and secondary minerals in between them have been intensively studied with XRD and wet chemical techniques. The examination of these disturbed samples gave rise to a large number of secondary mineral names, including mixed layer or interstratified miner- als. Many of the secondary minerals cannot be determined with cer- tainty by light microscopic techniques because they are either too small or not distinguishable as separate entities in larger weathering biotite grains. This discrepancy between light microscopy, XRD and wet

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chemistry is gradually becoming less serious with the introduction of modern submicroscopic techniques.

1.2. WEATHERING CAPABILITY

Biotite ranks fourth in the mineral stability series of Goldich (1938), i.e. after olivine (least stable), augite and hornblende. It is more weatherable than potash feldspar, muscovite and quartz. The mineral has the same place in Jackson's (1970) weathering sequence of clay-sized minerals, viz. number four after gypsum, calcite and hornblende, together with olivine, pyroxene and anorthite. Biotite has a weathering potential index of 7 to 32 (Reiche, 1950) computed from chemical data.

1.3. IMPORTANCE IN SOILS

Fresh biotite is a common mineral in various intermediate and acid igneous and metamorphic rocks, but relatively rare in effusive rocks. The mineral is also present as a detrital component in a number of sediments. Consequently, biotite can be found over larger areas. Fresh and weathered biotite is often present in soils which form on slopes from bedrock and is then found as individual minerals or in rock fragments. In many cases pseudomorphs, formed by a variety of secondary minerals are found.

Biotite provides nutrients for plants upon weathering by release of interlayered potassium and octahedral magnesium.

2. UNWEATHERED BIOTITE

2.1. MINERALOGICAL DESCRIPTION

2.1.1. Chemical composition

Biotite comprises a series of monoclinic minerals of the mica group that may be represented by the generalized formula : $K(Mg, Fe^{2+})_{3-1.5}$ $(Al, Fe^{3+})_{0-1} (Al_{1-1.5} Si_{3-2.5} O_{10}) (OH, F)_2$ (Tröger, 1969).

Individual minerals of the biotite series are distinguished by their Mg/Fe^{2+} ratio, viz. phlogopite (Mg 100/Fe 00), meroxene (Mg 80/Fe 20), lepidomelane (Mg 50/Fe 50), siderophyllite (Mg 20/Fe 80) and annite (Mg 00/Fe 100). The composition of the biotite series is, however, exceedingly variable because atomic substitution of Mg, Fe, Ti and Mn occurs. Fluor can substitute for some OH, and Na, Ca, Ba, Rb or Cs can partly replace K in the interlayer (Mason & Berry, 1968).

Oxybiotite is characterized by the oxidation of ferrous iron, and the substitution of some OH by oxygen. The general formula is $KMg Fe_2^{3+}$ $(AlSi_3O_{10}) O_2$.

2.1.2. Crystallographic data

Monoclinic system : 2/m; one polymorph is trigonal.
Cleavages : (001) perfect.
Habit : in hand specimen : platy, pseudohexagonal (001), (110), (010)
crystal faces. The mineral is flexible and elastic; in thin sections : euhedral
and anhedral. Buckling of the mineral may occur.

2.1.3. Optical properties (Mason & Berry, 1968, Tröger, 1971, Mackenzie & Guilford, 1981).

Optical orientation : a $\wedge$ z, b = y, c $\wedge$ X; optical plane is parallel to (010).

	Phlogopite	*Meroxene*	*Lepidomelane*	*Annite*	*Oxybiotite*
	$\longleftarrow$		Mg		
			Fe	$\longrightarrow$	
n_x	1.522	1.571	1.598	1.616	1.600-1.610
n_y	1.548	1.609	1.651	1.696	1.677-1.722
n_z	1.549	1.610	1.652	1.697	1.680-1.730
Δ	0,027	0,039	0,054	0,081	0,080-0,120
$2V_x$			$0°-35°$		$20-30°$
Opt. sign			B^-		B^-

Colour in thin sections : the colour of the biotite series varies between
yellow, brown, red-brown, blue-green and green. Phlogopite can be
colourless. Pleochroism may be present in yellow and brown, sometimes
green. Iron and possibly titanium content can affect the colour of bio-
tite. A mottled appearance becomes visible when the mineral is close to
an extinction position.

Oxybiotite is highly pleochroic, with colours varying from yellowish
brown to dark reddish brown.

Inclusions : inclusions are numerous, e.g. zircon, monazite, xenotime,
orthite, titanite, wolframite, apatite and rutile; some of them surrounded
by pleochroic halos. Saginite inclusions, frequently acicular rutile, are
common in biotite minerals.

2.2. OCCURRENCE

2.2.1. Igneous and metamorphic rocks

Phlogopite, the Mg-biotite, occurs in basic and especially ultrabasic
rocks such as mica-peridotite and kimberlite. It can be a constituent of
serpentinite and pyroxenite and also occurs in some marbles of contact
and regional metamorphism. Meroxene is found in gabbros, morites,
lamprophyres, basalts and amphibolites. Lepidomelane is the most
common biotite in rocks and found especially in granites, granodiorites

and their pegmatites, but also in quartzdiorites, tonalites and in many lamprophyres. Gneisses, mica schists and hornfelses also contain lepidomelane. Siderophyllite is mainly present in foyaite, syenite and aplogranite. No data on the occurrence of annite in rocks are found.

2.2.2. Sediments

Phlogopite is usually present in younger sediments. It is frequently replaced rapidly and is therefore relatively rare in older sedimentary deposits. Detrital biotite is often reported in descriptions of unconsolidated sediments. It occurs in arid regions and in sediments of rivers which erode biotite-containing bedrock. The mineral is common in certain sandstones, especially in greywacke.

2.3. MINERAL ASSOCIATIONS

Lepidomelane, the most common biotite, is generally associated with some of the following minerals : muscovite, quartz, alkali feldspars and acid plagioclases, epidote and hornblende, chlorite, apatite, monazite, xenotime, rutile, tourmaline, zircon, titanite, magnetite, etc.

Biotite and chlorite can often be observed together in the so called fresh rock. In such cases biotite is often more or less altered to light green chlorite. This alteration may follow two different patterns : along cleavages (parallel linear or banded pattern) or starting from the surface (pellicular alteration), (Plate I, a and b). Ultimately, the whole crystal may be replaced and a pseudomorph formed, e.g. in many lamprophyres. Hardly any precise information exists on this alteration type, its conditions or its products. The alteration to pennine and ripidolite is mentioned by Tröger (1969). This secondary chlorite has the same optical orientation as the original biotite, but may be distinguished from it by the green pleochroic colours, the very low birefringence, or the abnormal blue interference colours.

3. WEATHERING PRODUCTS OF BIOTITE

3.1. INTRODUCTION

Biotite can occur in a variety of soils and is rather easy to recognize in thin sections. In the initial phase of weathering it often weathers along cleavages and at the extremities of the lamellae. In between these lamellae one can find secondary minerals which have often lost the biotite structure, although their own structure may be influenced by the original biotite. Consequently a complicated set of secondary minerals may occur between the lamellae.

Various subdivisions can be made to present secondary minerals de-

rived from or associated with biotite. The one chosen here is related to the structure of biotite and the manner in which the secondary minerals formed. To obtain an understanding of the processes involved, light microscopic, XRD, wet chemical and submicroscopic techniques have been used. The following subdivision has been made for secondary minerals derived from biotite :

— secondary minerals which retained some of the original biotite structure, e.g. vermiculite and smectite (section 3.2);
— secondary minerals formed from solutions which had a composition strongly influenced by the weathering products of biotite, e.g. kaolinite, gibbsite, iron compounds and titanium oxides (section 3.3);
— secondary minerals which did not form at the expense of biotite, but which can be found in an interlamellar position of weathered biotite, e.g. calcite, gypsum (section 3.4). Secondary minerals such as kaolinite, gibbsite and iron compounds may also crystallize from external solutions.

3.2. SECONDARY MINERALS WHICH RETAINED SOME OF THE ORIGINAL BIOTITE STRUCTURE

3.2.1. The structure of unweathered biotite (fig. 1)

Biotite is a 2:1 phyllosilicate or layer silicate. It comprises planes of ions which can be combined to form sheets, viz. an octahedral sheet situated between two tetrahedral sheets. These three sheets form a 2:1 layer which, together with the interlayer cations (mainly K), represent the unit structure of biotite (Fanning & Keramidas, 1977).

The tetrahedral sheets consist of tetrahedra which are composed of four oxygen ions (at the apices) and Si or partially Al ions (in the centres). Three oxygen ions are shared with neighbouring tetrahedra and form a basal plane of anions on both sides of the biotite layer. The

Fig. 1.
Structure of biotite :
T : tetrahedral layer;
O : octahedral layer;
K^+ : interlayered potassium.

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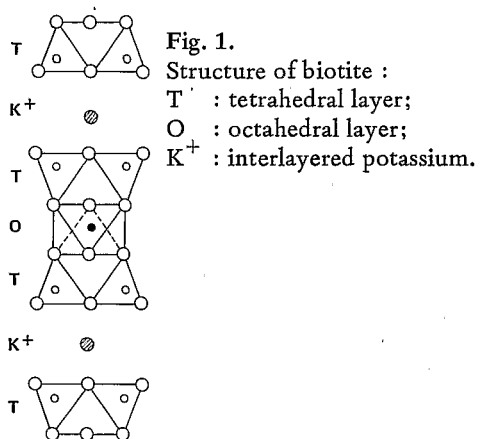
fourth oxygen ion of the tetrahedron is directed inwardly and shared with an octahedron of the octahedral sheet which forms the middle part of the 2:1 layer. The octahedron comprises six anions, viz. four oxygen ions at its apices which are shared with four tetrahedra of the tetrahedral sheet, two hydroxides in the two remaining apices, and a central Mg^{2+} or Fe^{2+} ion. Because 3/3 of the octahedral sites are occupied by divalent iron or magnesium, biotite is said to be a trioctahedral mineral. Muscovite, also a 2:1 mica, has only 2/3 of the central positions of the octahedra filled with predominantly trivalent cations (Al^{3+}) and is therefore a dioctahedral mica. The occupation of 3/3 or 2/3 of the centres of the octahedra and changes in composition of ions in the interlayer, play an important role in the formation of secondary minerals in which some of the original structure of biotite is retained.

3.2.2. Structure transformations during biotite weathering

The structural transformations during the weathering of biotite can partly be summarized as a loss of interlayer K which is compensated by an intake of hydrated exchangeable cations. In this way expansible 2:1 minerals are formed (Fanning & Keramidas, 1977). This is illustrated in figure 2. The transformation can take place by layer weathering (fig. 2b) or by edge weathering (fig. 2c).

In layer weathering some interlayers are "opened" throughout the crystal, this means that the K^+ is replaced by hydrated exchangeable cations. As long as not all interlayers are opened, various types of mixed layers or interstratified minerals are formed, e.g. biotite-vermiculite and biotite-smectite. Hydrobiotite is probably also a representative of this category of weathering biotite. In edge-weathering, biotite layers are opened along edges and fractures. Wedge-shaped openings can be found which force the layers apart. The core of the grain is initially unaffected. In this way *intergrades* are formed (e.g. intergrade biotite vermiculite).
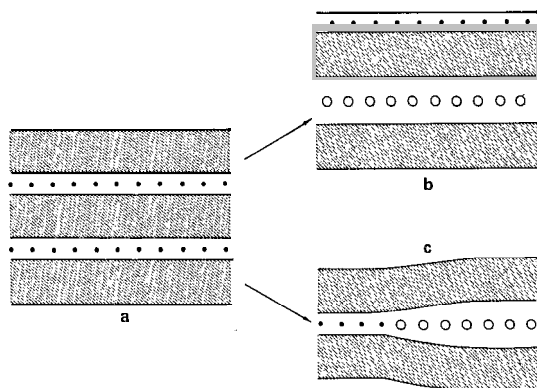

Fig. 2.
Schematic representation of biotite weathering : a : unaltered biotite, b : layer weathering, c : edge weathering.

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Eventually, when all interlayered K is replaced and the layer charge is superior to 1.2 per unit, the newly formed mineral is called vermiculite; when the charge is inferior to 1.2, it is called a smectite.

The weathering may also affect the tetrahedral and octahedral layers, e.g. oxidation of ferrous iron, exchange of ions. When Si increases and Al decreases in the tetrahedral sheets and K is lost from the interlayer, phlogopite alters to Mg-rich saponite (Borchardt, 1977). The transformation of the phlogopite to vermiculite and saponite has also been described by Sridhar & Jackson (1974). SEM studies of the lithium-saturated, electrical double-layer-swollen, freeze-dried macroflakes of phlogopite have revealed that saponite could glide out from interstratified sites within the mica, to fold at the mica-vermiculite grain boundary.

According to several authors (e.g. Jackson, 1963; Seddoh & Pedro, 1974) an hydroxy-Al interlayer may form under given conditions, transforming the vermiculite or smectite to a mineral called Al-chlorite, soil chlorite or hydroxy-Al interlayered vermiculite. Since this has only been described in clay fractions, and no exact optical micromorphological data are known, it will not be discussed in this paper. The same applies to Mg-chloritization (Wilson, 1970, 1975) which may occur if an excess of Mg is present, e.g. from neighbouring hornblende grains.

3.2.3. Optical characteristics

As the above mentioned transformations affect the biotite, a variety of intermediary minerals can be formed according to XRD data. With light-optical techniques only two end products can be recognized : vermiculite and smectite. For the intermediary stages (mixed layers and intergrades) the optical characteristics are not well defined.

Changes in optical properties characterizing the weathering of biotite to vermiculite or smectite refer to the gradual discolouring to pale yellow, the loss of pleochroism, the decrease in birefringence from second to first order, the decrease of the refractive indices and the disappearance of mottling near the extinction position (Walker 1949, Wilson 1966, Meunier 1977). According to Walker (1949) the optical angle 2V initially increases, but subsequently decreases; he also observed a diminution in the definition of the interference figure which is consistently biaxial negative. Wilson (1970) observed no definite change in 2V, whereas Bustin & Mathews (1979) advocate an increase in 2V from $0°$ in biotite to $20°$ in the weathering product.

Refractive indices of soil vermiculites are usually higher than those of hydrothermal vermiculite but no precise data are available. The optical characteristics mentioned below for vermiculite (Tröger, 1971) and smectite concern geological materials.
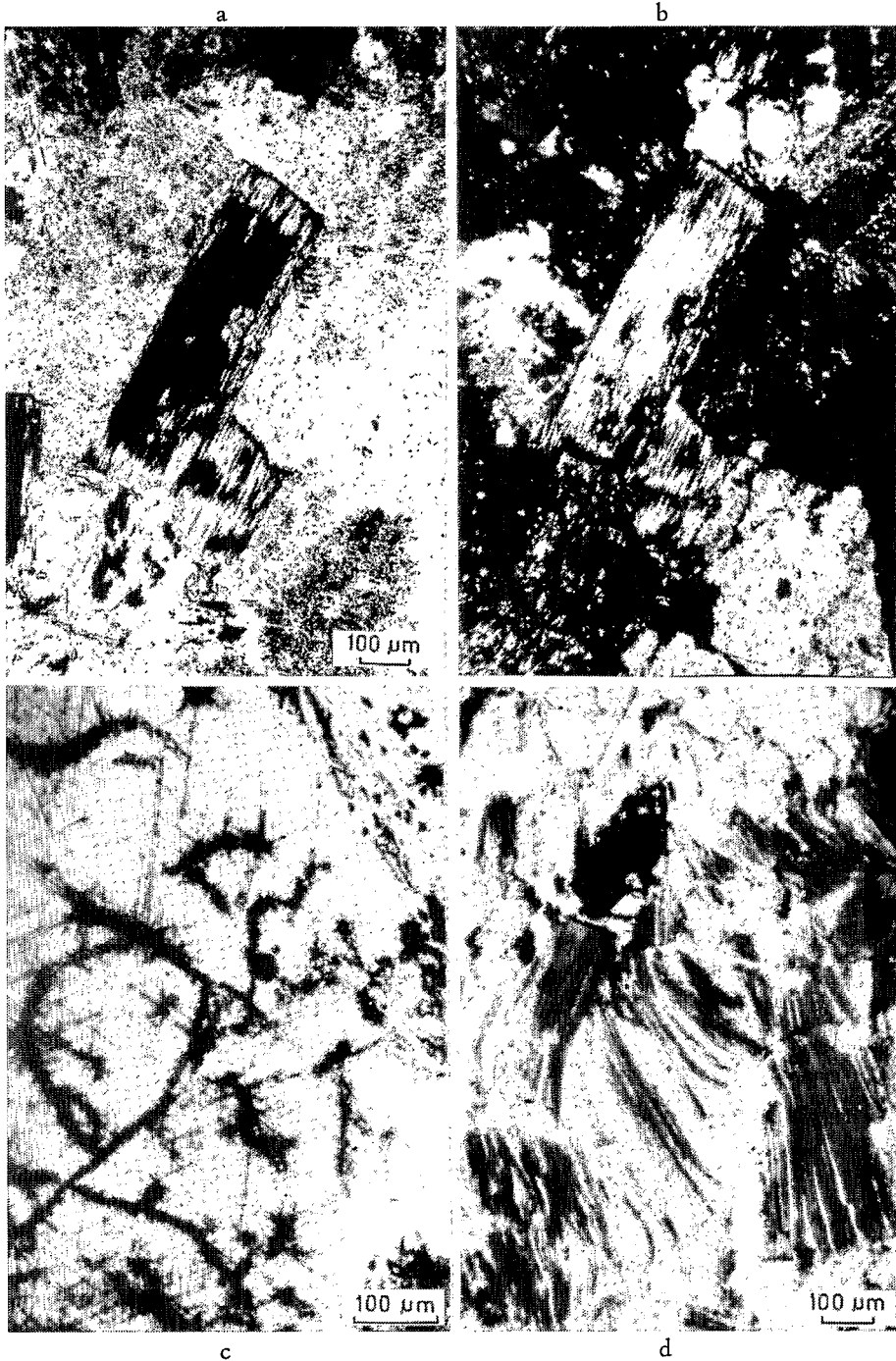
Plate I

a : Pellicular alteration of dark brown biotite (dark grey on micrograph) to pale greenish chlorite (light grey). Plain light.

b : idem, crossed polarizers.

c : Completely altered biotite into vermiculite with sagenite needles according to a trigonal pattern, parallel to (001). Plain light.

d : Exfoliated biotite (dark grey) with interlamellar montmorillonite (light grey). Plain light.

	Vermiculite	Montmorillonite	Saponite
n_x	1.525-1.561	1.475-1.503	1.48-1.54
n_y	1.545-1.581	1.499-1.533	1.50-1.58
n_z	1.545-1.581	1.500-1.534	1.50-1.58
Δ	0.020-0.020	0.025-0.031	0.02-0.04
$2V_x$	0°-8°	7°-25°	0°-40°
Opt. sign	B^-	B^-	B^-

Extinction of the pseudomorph : parallel.

Elongation of the pseudomorph : length low.

Colour in thin sections : colourless, over weakly green-yellow to weakly olive green, orange yellow to pale yellow or yellow-beige to colourless for vermiculite, and yellow-beige to pale yellow for smectite, with a weak pleochroism.

Optical characteristics determined on domains or clusters of clay-sized particules may deviate from the above mentioned data, because of factors such as form birefringence. This is especially the case for the smectite pseudomorphs.

Inclusions : some or most of the original biotite inclusions (e.g. rutile, apatite, monazite) may start to weather, whereas new inclusions e.g. of rutile, titanite or iron oxyhydrates may be formed as a result of the expulsion of these cations from the lattice (plate I.c).

The optical characteristics of smectites formed from weathered biotite are usually difficult or impossible to determine exactly because of their very small size and the variations in the refractive index of the impregnating material.

3.2.4. Micromorphology and submicroscopy

In the early stages of weathering, biotite often splits and opens out along cleavage planes (fig. 3A) and subsequently shows exfoliation parallel to (001) (Wilson, 1966). When internal stresses increase, the grain may break according to planes perpendicular or oblique to (001) (Bisdom 1967a). These microcrack systems often form part of larger ones along which rock fragments can eventually become detached from the bedrock (Bisdom 1967b). Such initial stages of biotite weathering frequently occur without discolouration and are possibly related to a K-release from the interlayer and a release of internal stresses. The microfracturation of the mineral may also occur under the influence of external stresses created for example by weathering and exfoliation of neighbouring biotite grains.

The next step in the weathering sequence of biotite shows an increase in the area which is affected by alteration processes. Curling, scrolling and buckling of the weathering biotite takes place. Wilson (1966)
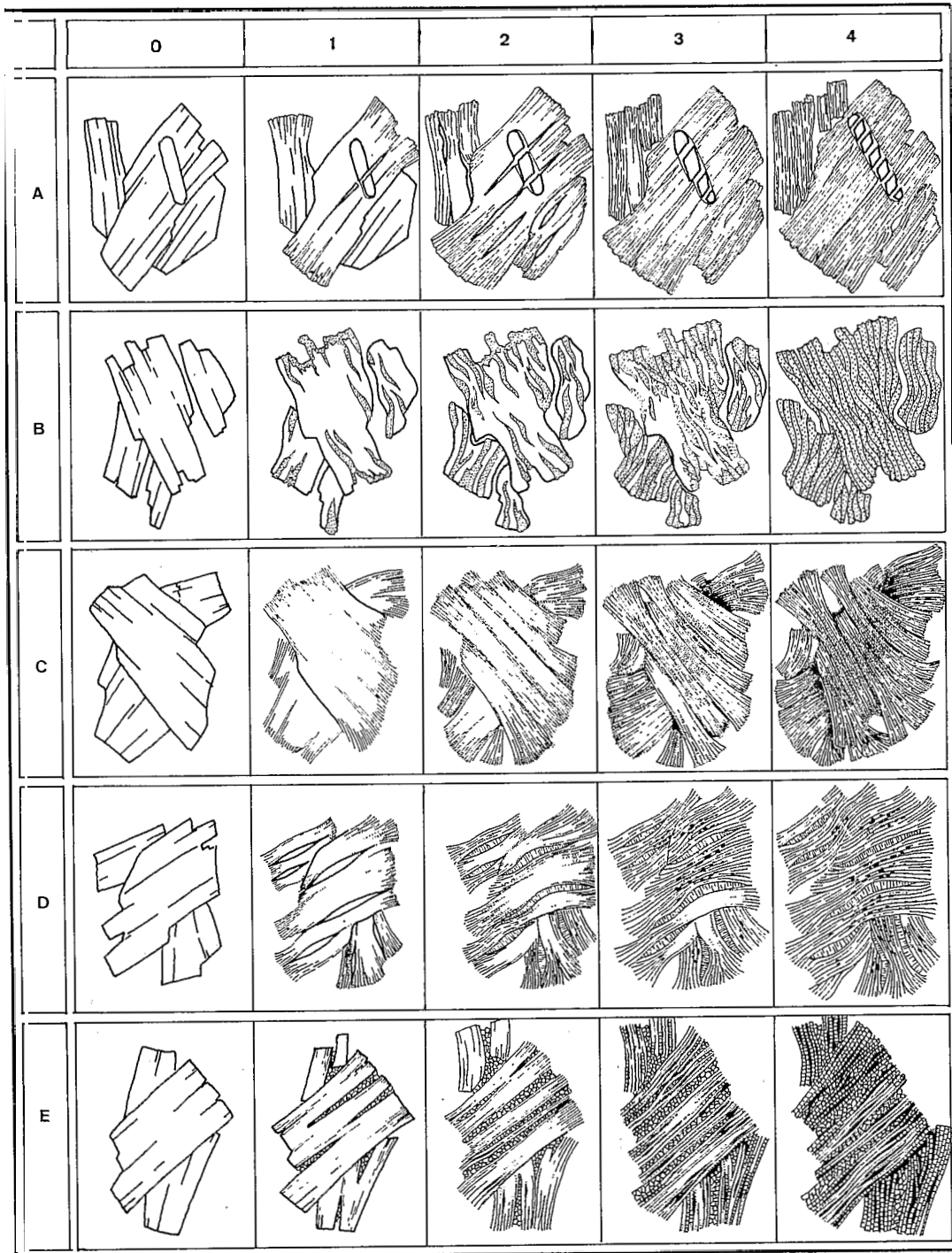
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Fig. 3.

Fig. 3.
Micromorphological aspects of biotite weathering.

The numbers of the vertical columns (0-4) refer to the degree of weathering as defined by Stoops et al. 1979.

A. Biotite to vermiculite. The weathering progressively affects tye crystal, starting from the boundaries (pellicular) or cleavages (linear). An important increase in volume, perpendicular to the biotite lamellae occurs which results in the fracturing and translocation of an included apatite crystal. Very fine newly-formed inclusions of iron and titanium compounds (dotted) are arranged between the vermiculite lamellae.

B. Biotite to smectite. The irregularly progressing weathering causes swelling and deformation of some lamellae, resulting in a katamorphous weathering type. This process is enhanced by the fact that the very weak and plastic smectite exhibits a tendency to flow and therefore to fill newly formed pores in the surrounding fractured and weathering minerals. Upon weathering, iron and titanium of the original biotite usually enter the smectite lattice and therefore do not form independent particles.

C. Biotite to kaolinite and iron oxyhydrates. The weathering front usually progresses regularly into the crystal and this gives a significant increase in volume. As a result of the created compaction forces, layers of kaolinite show a tendency to glide over each other. This deforms the pseudomorph, gives it a step-like aspect and promotes exfoliation. The iron of the biotite cannot enter the kaolinite lattice and therefore crystallizes as small inclusions between the kaolinite plates in the central part of the pseudomorph, or at its boundaries.

D. Biotite to kaolinite and sagenite. Microcrystalline kaolinite, probably of allochtonous origin, may crystallize perpendicularly to the separated biotite lamellae thus forming irregularly distributed veins and lenses. The remaining biotite can subsequently weather to macrocrystalline kaolinite, which is often parallel to the original cleavage. Titanium compounds occur as sagenite crystallites between the kaolinite plates, as do iron oxyhydrates.

E. Biotite to gibbsite and iron-oxyhydrates. Separation of biotite lamellae may produce pores which can be filled with gibbsite, probably of allochtonous origin. Biotite may weather to regular layers of kaolinite and subsequently to autochtonous gibbsite which crystallizes perpendicularly to the kaolinite plates. These two gibbsite types may remain recognizable, even after the complete disappearance of biotite and kaolinite. Iron-oxyhydrates may form opaque layers parallel to the cleavages, and are conserved even after complete transformation of the biotite to gibbsite. Kaolinite flakes may subsist inside iron lamellae because they are protected.

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observed curled edges, surface irregularities, microcracks, cracks forming angles of 120° and scrolling in loose flakes. Such phenomena can also be found in thin sections and this may give considerable differences in the orientation of weathered and unaltered parts of the biotite flake.

Release of octahedral Fe may result in a bleaching of the grain if the iron is evacuated as a ferrous ion (plate I, d). When it is oxidized however, the insoluble ferric iron may precipitate between the lamellae and will result in opacity.

SEM was used by Tarzi & Protz (1978) to study splitting, exfoliation, crumbling and cracking of naturally weathered micas in loose samples. The edges of weathered biotite in the 250-500 μm fraction were very irregular, e.g. slanted or steplike. The (001) surface showed many holes, cracks and depressions. Splitting started along a few planes and reached the exfoliation stage by further separation of the microlayers during more advanced stages of weathering. Bustin & Mathews (1979) also studied weathering biotite with SEM and noticed expansion along cleavage planes and folding of the mineral edges.

Microchemical data on weathering biotite, obtained with submicroscopic techniques, were published by Seddoh and Pedro (1974, 1975), Meunier (1977) and Curmi (1979). EMA measurements indicated a decrease in K in the interlayer of weathering biotite flakes. Iron, released after oxidation of Fe^{2+} in the octahedral sheet, may be deposited along microcracks and cleavages in the biotite flake. In situ microchemical analysis of such iron compounds frequently shows a lower iron content than expected from light optical observations.

The heterogeneity of weathering biotite was measured with EMA by Meunier (1977). The Si/Al ratio became more variable after biotite was subjected to hydration and a loss of interlayer K could be demonstrated. Curmi (1979) conducted similar studies and found that the weathering biotite lost mainly Fe, K and Mg while Si and Al remained (table 1, plate III, b). Al could even become somewhat more concentrated. SEM-EDXRA measurements, done by Bisdom for this study, showed that the peak height of Al, compared to Si, changed most in weathering biotite. Smaller quantities of iron were measured in places where light microscopy indicated discolouring of the biotite.

SEM studies were combined with thermal neutron activation by Lee et al. (1974) to examine fission particle tracks in micas and micaceous vermiculite. The study demonstrated that voids created in this manner could affect the weathering process by influencing the diffusion between the sheets, layers and interlayers along the broken edges of vermiculite. Tarzi & Protz (1978) discussed similar voids in biotite, but interpreted them as dissolution holes from former inclusions.

Isomorphous or mesomorphous transformations of biotite to smectite

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Table 1

Point analysis (weight % of oxides) of a biotite with interlamellar kaolinite and exfoliations of vermiculite*

	Biotite	Vermiculite	Kaolinite
SiO_2	35.86	47.20	43.68
Al_2O_3	20.24	34.89	36.62
TiO_2	2.92	0.29	0.01
$FeO^{(1)}$	21.09	2.15	1.61
MgO	7.21	2.06	0.00
K_2O	8.23	2.06	0.14
CaO	0.00	0.00	0.20
Na_2O	1.08	0.77	0.00
Cr_2O_3	0.20	0.11	0.08
MnO	0.21	0.03	0.01
NiO	0.04	0.10	0.19
$H_2O^{(2)}$	2.86	10.34	17.45
SiO_2/Al_2O_3	3.01	2.30	2.03

 * After Curmi 1979
(1) Total iron expressed as % FeO
(2) Estimated by difference
Experimental conditions : EDS spectrometer, 15kV, 2,5nA.

have been observed in granite weathering under conditions of restricted drainage (Delvigne, unpublished). The smectite lamellae have approximately the same size as the original biotite grain and, because the cleavage planes are also concordant, the impression is that each biotite crystal has been replaced by a single smectite crystal. Its colour varies from very pale creamy yellow to beige-yellow, with a very weak pleochroism. Interference colours correspond to those of talc or sericite.

Two successive stages can be recognised in the weathering of biotite to smectite : oxidation of the iron which is subsequently expelled from the lattice and acts as a pigment (fine brown punctuations, partly masking the optical properties of the grain), followed by a bleaching of the grain as a result of the dissolution and extraction of the iron (plate I, d). The smectite may appear at the borders of the biotite grain (peripheral weathering) or as irregular interlamellar layers (parallel linear or banded weathering, fig. 3B).

The newly-formed smectite seems relatively unstable. Along the pores, the pseudomorph is observed to be subject to a mechanical breakdown : very gradually, small smectite fragments are carried away. Smectite can also be transformed to kaolinite without an apparent relation between the optical orientation of the smectite and that of the newly formed kaolinite.

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3.3. SECONDARY MINERALS FORMED FROM SOLUTIONS WHICH HAD A COMPOSITION THAT WAS STRONGLY INFLUENCED BY THE WEATHERING PRODUCTS OF BIOTITE

3.3.1. Biotite altering to kaolinite and halloysite

Kaolinite and halloysite are 1:1 phyllosilicates; $Al_4Si_4O_{10}(OH)_8$ and $Al_4Si_4O_{10}(OH)_8 \cdot 2H_2O$ respectively. Kaolinite is triclinic and appears as pseudohexagonal platy crystals in SEM and TEM. Sometimes, stacks of kaolinite platelets are observed. TEM and SEM micrographs of halloysite show slender hollow tubes.

Optical characteristics for kaolinite (Tröger, 1971) are :

n_x	1.553-1.565
n_y	1.559-1.569
$n_z 1$	1.560-1.570
Δ	0.007-0.005

The average refractive index for fine grained kaolinite ranges between 1.56 and 1.57, and that of halloysite between 1.528 and 1.542. Kaolinite booklets are length slow with respect to the cleavage planes. Both have first order grey interference colours, and in thin sections appear to be colourless, unless stained by iron oxyhydrates. They readily absorb safranine, from which they obtain a reddish staining. When safranine is applied to uncovered thin sections, the reddish kaolinite is pleochroic, whereas this is not the case for halloysite.

The alteration of biotite to kaolinite may occur due to hydrothermal agents, but is usually a result of weathering processes in soils, saprolites and saprocks (weathered, but still cohesive rock, Bisdom, 1967a).

Kaolinite can be observed to form in various stages of biotite weathering between exfoliated biotite and vermiculite lamellae (plate IIa, b). Light microscopic observations show that the kaolinite either oriented parallel (e.g. Wilson 1966, Bisdom 1967a) or perpendicular (Seddoh & Robert, 1972; Meunier, 1977; Curmi, 1979) to the walls of the adjacent weathering biotite lamellae (plate III, b). Epitaxial crystallization of the kaolinite, whereby the weathering biotite lamellae can act as templates which may control the orientation of the kaolinite during its growth was favoured by Wilson (1966) and Bisdom (1967a). This type of crystallization may have affected the parallel and perpendicular growth of kaolinite in between weathering biotite lamellae. Gilkes & Suddhiprakarn (1979a) favoured both epitaxial and topotaxial crystallization of kaolinite, whereby topotaxial crystallization means a displacive transformation of biotite to kaolinite in which primary interatomic bonds may remain in the newly-formed kaolinite.

When the alteration of the biotite lamellae proceeds, one often observes that they bend and buckle (fig. 3C; plate III, d). The result is that kao-
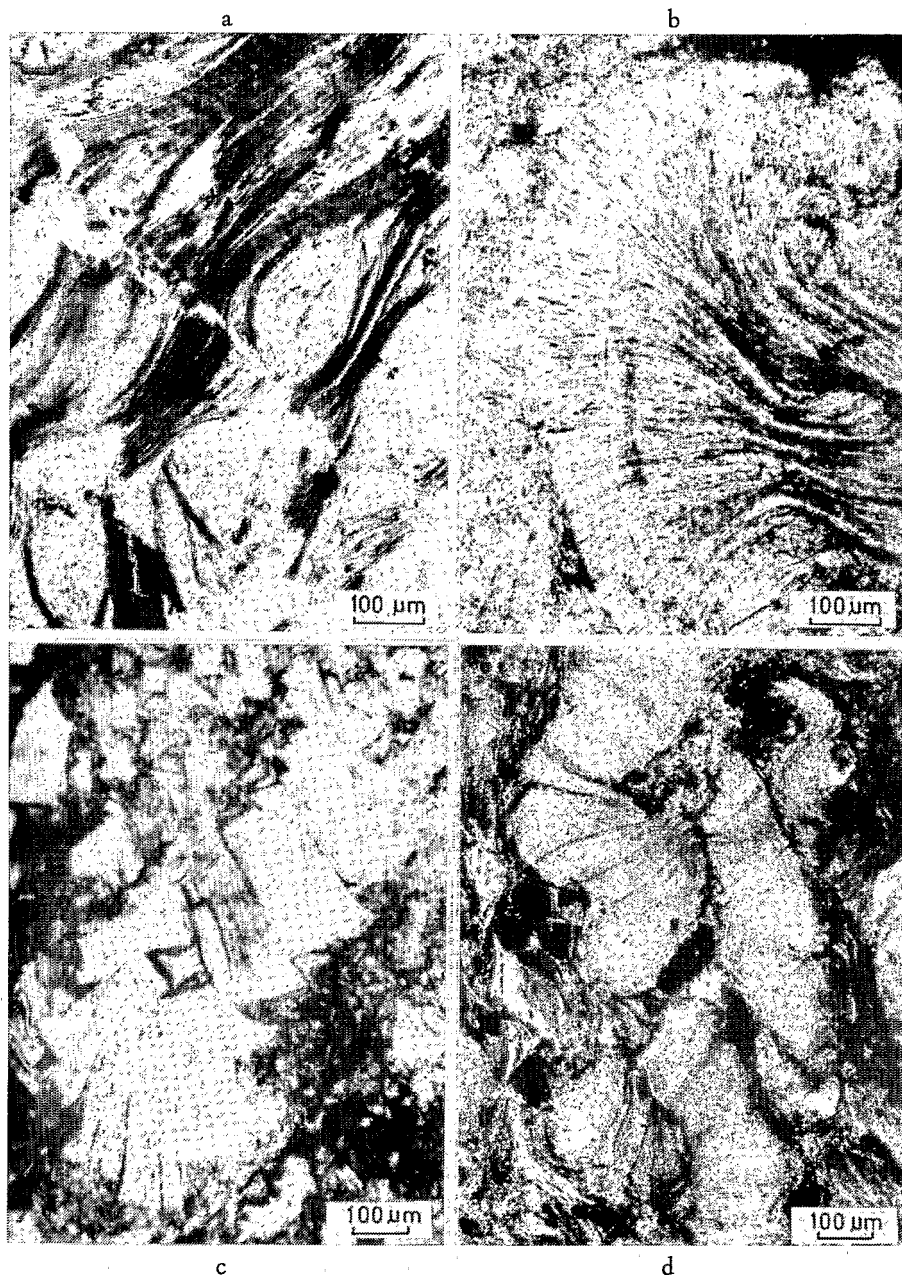
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Plate II

a : Exfoliated and curled weathering biotite (dark grey) and interlamellar cyrstalliza-
tion of kaolinite (light grey). Minute opaque droplets containing Ti occur in the
kaolinite. Plain light.

b : Sheaf-like or brush-like exfoliation of biotite (dark grey) weathering to kaolinite
(light grey) with numerous opaque Fe-Ti-particles. Plain light.

c : Final stage of biotite weathering into kaolinite with exfoliation and dislocation
according to (001). Plain light.

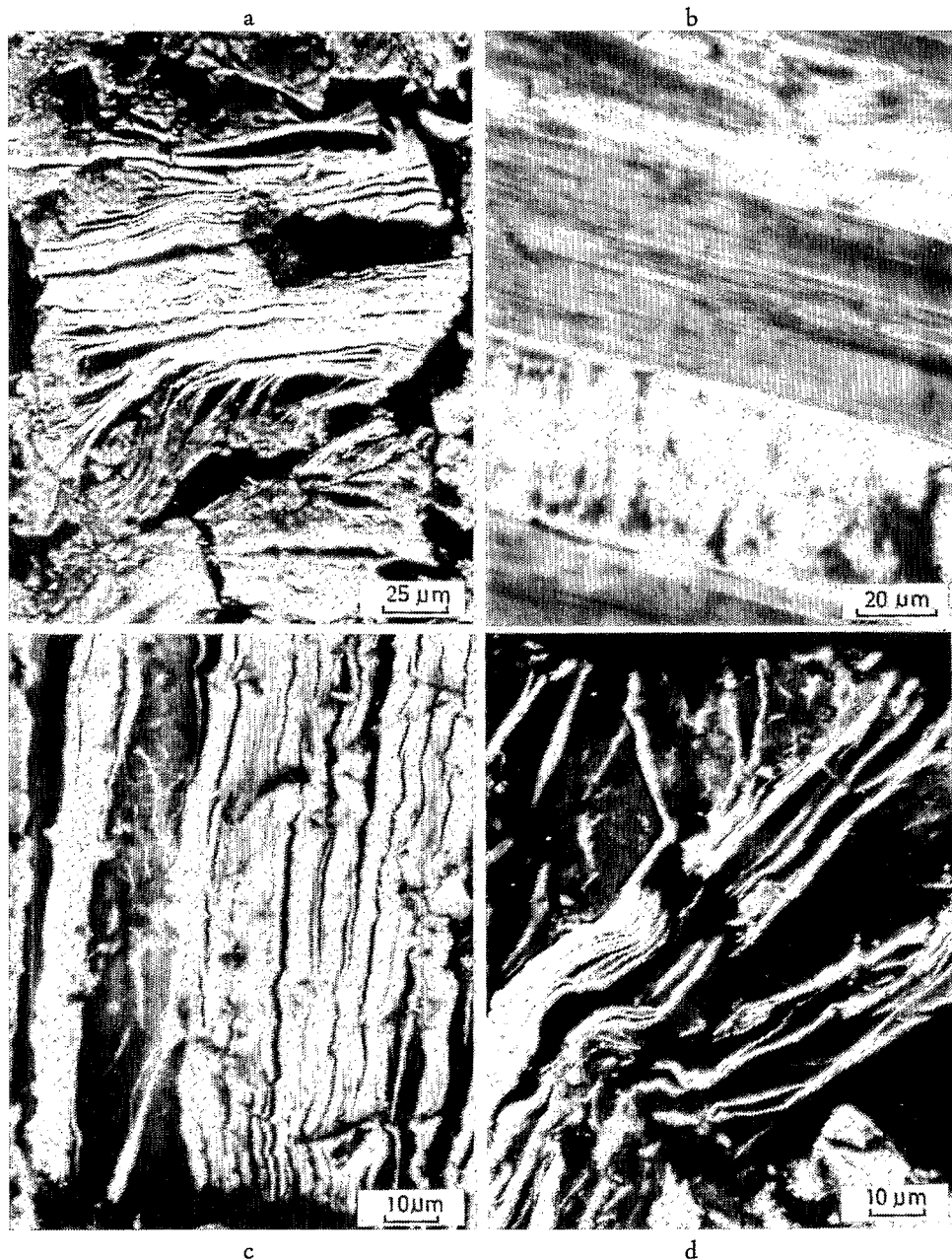d : Kaolinite vermiforms after biotite with intercalated Fe-oxyhydrate particles.
Plain light.

Plate III

a : Exfoliated weathering biotite with brush-like extremities and locally with inter-
lamellar kaolinite. Backscattered electron scanning image (BESI) on thin section.

b : Biotite showing perpendicularly oriented interlamellar kaolinite (below) and
vermiculite exfoliation (above). Point analysis on these minerals are reported in
table 1. Crossed polarizers.

c : Interlamellar crystallized kaolinite between exfoliated weathered biotite (left)
showing various orientations. Exfoliation and buckling of individual biotite-
vermiculite lamellae. BESI on thin section.

d : Exfoliated and buckled biotite-vermiculite lamellae partly with disoriented inter-
calated kaolinite. BESI on thin section.

linite in between these curbed lamellae may exhibit less and less orientation with respect to the weathering biotite lamellae (plate III, c). Conversely, crystallizing kaolinite will also affect the orientation of the weathering adjacent biotite lamellae.

If no topotaxial crystallization of kaolinite occurs in weathering biotite, the primary mineral is broken down completely before kaolinite can form. This weathering of biotite to kaolinite possibly occurs via a phase of amorphous aluminium-silicates (Wilson, 1966; Hsu, 1977).

If the weathering occurs in situ and without disturbance of the regolith, the isomorphous or mesomorphous transformation of biotite may give rise to large kaolinite books or vermiforms, described by many authors (plate II, c and d; fig. 3C). These vermiforms can be distinguished from those which originated in the saprolite and have no relation to weathering biotite. Pseudomorphs after biotite are much larger than the authigenous kaolinite vermiforms. The vermiforms may contain Ti compounds with various forms and degrees of crystallization (plate IV, a).

If iron, which was expelled from the biotite lattice, was not removed from the grain in early stages of weathering, interlamellar crystallization of iron oxyhydrates could take place. If the thin sheets of goethite or hematite subsisted during the kaolinization process, kaolinite vermiforms were stained reddish or brownish.

Edge weathering of biotite grains is commonly observed during the kaolinization process. Wedge-shaped kaolinite zones penetrate the weathering biotite, produce exfoliation, and are responsible for the minerals sheaf-like shape (fig. 3C; plate III, a). The centre of the grains and lamellae between which kaolinite is present may consist of unaltered biotite in this stage of weathering. This irregular increase in volume affects the original rock fabric (Verheye & Stoops, 1975).

Completely weathered minerals, mostly katamorphous alterations, have been called minphantosepic by Bisdom (1967a). In relation to mica weathering the terms minphyllasepic and minphyllsepic were introduced. No orientation is observed with a light microscope in the secondary minerals, including kaolinite, between biotite lamellae in minphyllasepic fabric (min = plasmic fabric types of Brewer occurring inside a weathering mineral; phyll from phyllosilicates; and asepic as defined by Brewer, 1964). Orientation of secondary minerals can be present in minphyllsepic fabrics of weathering biotite (sepic as described by Brewer, 1964).

Various secondary minerals can be found inside one weathering biotite grain, indicating the diversity of microenvironments which exist in the altering biotite flakes (Penven et al., 1981). Other interesting data are from measurements which suggest that not all stages of weathering need to be present before kaolinite is formed from biotite. De Kimpe & Tardy (1968) indicated that weathering of biotite to kaolinite may occur

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without an intermediate vermiculite phase. Gilkes & Sudhiprakarn (1979) suggested that too high potassium levels in the groundwater may suppress the vermiculite phase.

Curmi (1979), using SEM-WDXRA measured SiO_2/Al_2O_3 ratios near 2 for kaolinite, 2.3 for exfoliated vermiculite and 3 for biotite (table 1, plate III, b). SEM-EDXRA measurements by Bisdom for this paper indicated that the peak height of Si in point analyses remains relatively constant during biotite weathering, whereas that of Al increases. When the transformation to kaolinite was virtually completed, both peaks had practically the same height. Most other elements, except Fe and Ti, were lost in EDXRA measurements of kaolinite.

Transformation of biotite to *halloysite* was observed by SEM in samples of the deeper part of a saprolite on gneiss in Malaysia by Eswaran & Yew (1976) and on granite in Algeria by Penven et al. (1981). Kabata-Pendias & Ryka (1968) reported the transformation of biotite to septe-amesite (a Mg-Al silicate with kaolinite structure) in the Paleozoic weathering of a biotite-gneiss. In the first stage of biotite weathering inter-layers of septe-amesite are emplaced. These increase in volume until a complete pseudomorph is produced. The weathering sequence of biotite to septe-amesite is characterized by a decrease of n_y (from 1.602 to 1.582), a decrease in birefringence (from 0.040 to 0.017), an increase of 2V (from 15° to 21°) and a change in optical character (from negative to positive). The presence of amesite in weathering biotite was also noticed by Sousa & Eswaran (1975).

3.3.2. Biotite altering into gibbsite

Gibbsite, $Al(OH)_3$, is monoclinic and has a perfect (001) cleavage. The $n_x = 1.566-1.568$, $n_y = 1.566-1.568$ and $n_z = 1.587-1.589$. The colour is frequently greyish to white in thin sections. It has a higher birefringence than quartz and kaolinite and a higher relief. Gibbsite is usually somewhat lath-like or may form radiating patterns consisting of individual gibbsite crystals. Interpenetration twinning is frequent. The mineral may also occur as encrustations.

Gibbsite has often been reported in weathering biotite (e.g. Wilson, 1966 and 1975, Sidhu & Gilkes, 1977). In general the crystals are oriented perpendicular to the biotite lamellae, but oblique orientations have also been observed (fig. 3E, plate IV, c). It is not known with certainty whether Al is derived from the biotite lattice itself or from out-side sources. Gibbsite and kaolinite may already occur in early stages of biotite weathering between exfoliated lamellae.

3.3.3. Titanium and iron new-formations during biotite weathering

Both Ti and Fe occur in the crystal lattice of biotite or as inclusions,

and are frequently found in its weathering products, even after the biotite has disappeared completely. Anatase, goethite and amorphous titanium and iron compounds are the most important secondary products.

In several cases, rutile needles, arranged according to a regular hexagonal pattern are observed in altered biotite (plate I, c); they may be primary inclusions present in the biotite, or secondary minerals formed during biotite weathering. In other cases, especially in biotite, with rutile and sagenite inclusions, one can often observe the formation of minute droplets which may subsequently unite to form a turbid type of anatase (Bisdom, 1967a) (plate IV, b). This secondary anatase is easily recognizable in thin sections by its high relief, high birefringence and greyish amorphous to semi-crystalline appearance. The composition of such anatase and of the droplets was determined using SEM-EDXRA and LAMMA (Bisdom et al., 1981). The Ti content seemed to increase with cristallinity. Significant quantities of Al, Si and Fe were present besides a number of trace elements in the droplets and the secondary anatase. Craig & Loughan (1964) found that Ti can be transported over varying distances dependent on the pH of the environment. Ti is usually released from the parent mineral as $Ti(OH)_4$ which can precipitate to form an amorphous hydrous oxide gel. The gel can subsequently dehydrate to form a granular aggregate of small anatase crystals.

When the Ti compounds occur, together with vermiculite, kaolinite, or halloysite, at the site of a completely or partly altered primary biotite, one can often recognize a pattern which roughly coincides with the interlamellar positions of secondary minerals in the weathering biotite. However, the lamellae of biotite are frequently bent strongly during weathering, and this may also be the case as far as the secondary Ti accumulations are concerned. Such patterns of Ti compounds can often be used as a marker for completely weathered biotite (Bisdom, 1967a).

EMA studies by Jepson & Rowse (1975) of kaolinite showed that some 12 per cent of the Ti found by conventional chemical analysis was associated with this clay mineral, i.e. as Ti-oxide or as an isomorphous substituent. Adams et al. (1976) pointed out that both Ti and Fe may be present in kaolinites as discrete mineral species or in the structure of kaolinites where they substitute for Al and Si.

Iron oxyhydrates are frequently accumulated in various positions of weathering biotite after oxidation of ferrous iron in the octahedral layer of biotite and subsequent ejection as ferric iron. They are often present as yellowish brown-orange deposits in microcracks, between cleavages and on the surface of weathering biotite (e.g. Meyer & Kalk, 1964). Detailed studies of iron accumulations in weathering biotite (Gilkes & Suddhiprakarn, 1979a & b) were done with XRD, submicroscopy and light microscopy. Micrographs demonstrated that goethite could be
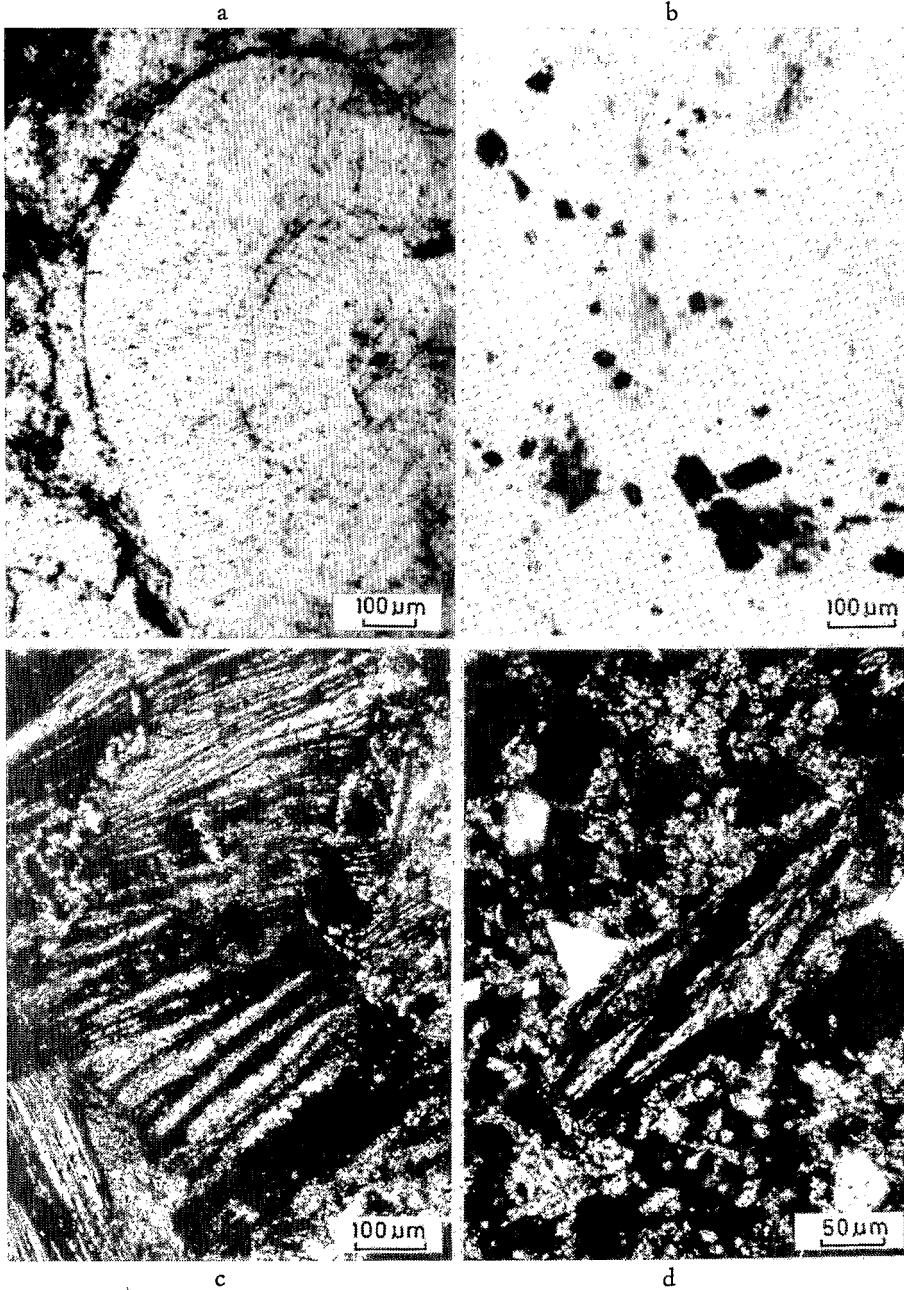
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Plate IV

a : Vermiform of kaolinite after biotite with inclusions of Fe and Ti containing drop-
lets. Plain light.

b : Secondary anatase in kaolinite matrix after katamorphous weathering of biotite.
Plain light.

c : Biotite weathered to iron oxyhydrates (opaque) with interlamellar crystallized
gibbsite (white). Crossed polarizers.

d : Weakly weathered biotite with interlamellar crystallized calcite. Crossed polar-
izers.

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present as aggregates in the weathering biotite flakes, as hexagonally oriented lath-like crystallites or as discrete particles. Electron diffraction patterns indicated that the close packed anion layers in goethite coincide with the brucite-like layer of micaceous minerals. The c- and b-axes of goethite were parallel with respectively the b- and a-axes of biotite. Epitaxial growth, however, was excluded by the authors.

Sousa & Eswaran (1975) described the pseudomorphous alteration of biotite to goethite in a saprolite from Angola. Microdroplets (framboids) of secondary goethite covered the surface of the weathering biotite flakes and were also observed between the exfoliated layers. XRD indicated the presence of kaolinite, amesite and amorphous compounds. Jackson et al. (1973) demonstrated the presence of Fe and Al hydroxide-crusts on intermittent cleavages of micaceous vermiculite macroflakes with SEM.

After complete weathering of the biotite, an alternation of goethite and gibbsite layers may sometimes be observed in thin sections (plate IV, c). The deposition of iron oxyhydrates in and around weathering biotite grains in a saprolite may result in the formation of primary ferruginous mottles (Fölster et al., 1971).

3.4. SECONDARY MINERALS WHICH DID NOT FORM AT THE EXPENSE OF BIOTITE, BUT WHICH CAN BE FOUND IN AN INTERLAMELLAR POSITION OF WEATHERED BIOTITE

Secondary minerals may form inside weathering biotite from solutions which entered the mineral and contain chemical elements which were not present in the original mineral. Examples are calcite (plate IV, d) and gypsum which are sometimes found in exfoliated biotite flakes (in more arid climates). Other secondary minerals such as goethite, gibbsite, chalcedony and kaolinite may form from components of introduced solutions, but can also form from weathering products of the biotite itself. Most of the amorphous weathering products inside altering biotites studied by Bisdom with submicroscopic techniques, contained mainly Fe, Ti, Al and Si.

4. CONCLUSIONS

A number of weathering sequences are known for biotite. Various intermediary secondary minerals have been named which differ only partly in their lattice formation from biotite. Such changes can occur in the interlayer of biotite (removal of K) or affect the cations of the tetrahedra or octahedra (removal, oxidation), resulting in the formation of interstratified mica-vermiculite, vermiculite, smectite, Al-chlorite, etc. Such knowledge, however, is mainly derived from XRD analysis and only

some of these secondary minerals can be recognized with the light microscope. Even when the more common submicroscopic techniques are used, such differentiation in secondary minerals as given by XRD analysis cannot be confirmed. It is likely, however, that in the near future electron diffraction techniques by STEM (scanning transmission electron microscope) can be used for additional determinations on a combination of loose materials and ultrathin thin sections.

The combination of light microscopy and submicroscopy allows in situ microchemical analysis of various weathering stages of biotite. Al and Si usually remain upon weathering, although these too may be transported away. In most cases, biotite weathers to vermiculite or smectite before kaolinite or gibbsite is formed although this does not seem to be essential.

Light microscopical studies of weathering biotite flakes often demonstrate that various secondary minerals can be present in one exfoliated flake, differing significantly from secondary minerals expected to form using knowledge from chemical measurements of the general soil environment. This can be explained by taking a wedge as an example. Where the two legs coincide the solutions meet the smallest part of the wedge and are unable to move freely; consequently the weathering products of the biotite will significantly determine the composition of the secondary minerals or amorphous materials formed at such places. At the opening of the wedge, however, solutions can move freely and soil solutions are thought to play a dominant role in the new-formation of secondary minerals.

As a consequence of the transformation of biotite to clay pseudomorphs, an important increase in volume is noticed especially parallel to the c-axis. This deforms the original outline of the grain and may disturb the original rock fabric. Large biotite flakes may weather to kaolinite vermiforms of sand or even larger sizes. Ti accumulations are often present in such vermiforms and frequently indicate sites of former cleavages in the weathering biotite. During the kaolinization of biotite both mesomorphous and katamorphous alterations may take place with changes in external shape and dimension. Kaolinite which crystallized from solutions and which was not related to biotite weathering may also exhibit vermiforms. However, these can be distinguished by a much smaller size, regular form and a lack of biotite-derived inclusions.

Ti frequently remains after the biotite has weathered in which case it is often found in thin sections as cloudy amorphous accumulations and secondary anatase. Fe compounds (e.g. goethite) are also found at the weathering sites of biotite, but it is often difficult to know whether the iron was generated inside the weathering mineral or precipitated from solutions introduced in the altering biotite; unless specific distribution

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patterns are evident.

To allow a detailed analysis of the processes and products involved in biotite weathering, a combination of various techniques is necessary. Most frequently XRD and wet chemical techniques are used for loose samples and in situ analysis on thin sections is done with the light microscope and submicroscopy. The combination of these techniques has allowed significant progress in obtaining a better understanding of weathering processes which affect the altering biotite mineral.

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Summary

   Three types of secondary minerals are distinguished in biotite weathering : (i) minerals which retained part of the original biotite structure e.g. vermiculite, smectite, (ii) minerals formed from solutions which had a composition strongly influenced by the weathering products of biotite e.g. kaolinite, gibbsite, iron compounds and titanium oxides, and (iii) minerals which did not form at the expense of biotite,

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but can be found in an interlamellar position e.g. calcite, gypsum. Secondary minerals such as kaolinite, gibbsite and iron compounds may also crystallize from external solutions.

Weathering of biotite to vermiculite and smectite is a gradual transformation involving several intermediary products (mixed layer minerals and intergrades) which can be recognized by XRD and chemical analyses, but have no well-defined optical characteristics.

Kaolinite pseudomorphs after biotite can be formed directly or through intermediary stages and different morphological and genetic types may be distinguished. Fe and Ti released during weathering can crystallize in situ and give rise to different types of inclusions or incrustations.

Emphasis is placed in this publication on light microscopic observations and interpretations of weathering biotite in thin sections. In situ submicroscopic data from electron microscopy were incorporated.

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Micromorfologie van de biotietverwering en van de secundaire mineralen

Samenvatting

Bij de verwering van biotiet onderscheidt men drie soorten secundaire mineralen : (i) mineralen die de biotietstruktuur ten dele behielden, zoals vermikuliet en smektiet, (ii) mineralen die ontstonden uit oplossingen waarvan de samenstelling sterk beïnvloed werd door de verweringsprodukten van biotiet, zoals kaoliniet, gibbsiet, ijzer en titaniumverbindingen, en (iii) mineralen die niet ten koste van biotiet gevormd werden, maar die tussen zijn bladen kunnen worden aangetroffen, zoals calciet, gips. Secundaire mineralen zoals kaoliniet, gibbsiet en ijzer oxyhydraten kunnen ook rechtstreeks ten koste van uitwendige oplossingen ontstaan.

De verwering van biotiet tot vermikuliet en smektiet is een geleidelijke omzetting, waarbij verschillende tussenprodukten gevormd worden (geïnterstrafiëerde mineralen en overgangsvormen) die kunnen geïdentificeerd worden met Röntgendiffraktie en chemische analysen, maar die geen wel bepaalde optische kenmerken hebben. Kaoliniet pseudomorf naar biotiet kan direkt of via tussenstadia gevormd worden, en verschillende morfologische en genetische typen kunnen onderscheiden worden. Het ijzer en titaan, vrijgekomen tijdens de verwering, kan in situ uitkristalliseren en aanleiding geven tot verschillende typen van insluitsels en korsten.

In deze publikatie is de nadruk gelegd op lichtmicroscopische waarnemingen en interpretaties van verwerende biotiet in slijpplaten. In situ submicroscopische gegevens van elektronenmicroscopie werden eveneens opgenomen.

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Micromorphologie de l'altération de la biotite et des produits secondaires

Résumé

Trois types de minéraux secondaires peuvent être distingués dans la biotite altérée : (i) ceux qui conservent en partie la structure de la biotite, comme la vermiculite et la smectite, (ii) ceux qui sont formés à partir de solutions dont la composition est fortement influencée par les produits d'altération de la biotite, comme la kaolinite, la gibbsite et des oxydes et hydroxydes de fer et de titane, et (iii) ceux qui ne se forment pas aux dépens de la biotite, mais qui peuvent être observés en position

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interlamellaire, comme la calcite et le gypse. Certains minéraux secondaires, comme la kaolinite, la gibbsite et les oxyhydrates de fer peuvent se former aussi à partir d'une solution externe.

L'altération de biotite en vermiculite et smectite est une transformation graduelle comportant différents produits intermédiaires (minéraux interstratifiés et intergrades) qui peuvent être déterminés par la diffraction aux rayons X et l'analyse chimique, mais qui n'ont pas des caractères optiques bien déterminés.

Des pseudomorphoses de biotite en kaolinite peuvent se former soit directement soit par des phases intermédiaires, et différents types morphologiques et génétiques peuvent être distingués. Fe et Ti libéré lors de l'altération peuvent crystalliser in situ et donner naissance à des types différents d'inclusions et d'incrustations.

Cette publication insiste principalement sur les observations faites en microscopie optique et sur les interprétations de l'altération de la biotite à partir de plaques minces. Y figurent également des résultats d'analyses submicroscopiques obtenues au microscope électronique.

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