

WEATHERING OF A CHROMIAN MUSCOVITE TO KAOLINITE

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Abstract—Single crystal X-ray diffraction and electron-optical analysis were used to investigate the weathering of a chromium-bearing muscovite (fuchsite). The muscovite had mostly altered to kaolinite with minor amounts of halloysite occurring between kaolinite plates. Evidence for both epitactic and topotactic growth of kaolinite from muscovite was obtained and no intermediate poorly-crystalline phases were detected. About half of the Cr in fuchsite was incorporated into kaolinite, whereas most of the Ti, Fe and Mg was lost.

Key Words—Alteration, Chromian muscovite, Kaolinite, Topotaxy, Weathering.

INTRODUCTION

Weathering of mica in soils has been investigated by many workers, with most research being directed towards biotite (Meunier and Velde, 1979; Gilkes and Suddhiprakarn, 1979; Ahn and Peacor, 1987; Banfield and Eggleton, 1988). In contrast, weathering of muscovite, which is a common constituent of soils, has received relatively little attention (Banfield and Eggleton, 1990). The weathering of muscovite is expected to differ from that of biotite in several respects as the chemical compositions of the octahedral and tetrahedral sheets are similar to those of the more common clay minerals (kaolinite, halloysite, illite). Therefore, relatively small structural and chemical modifications of the muscovite structure are required for its transformation to these clay minerals. Consequently, the weathering mechanism of muscovite and the retention of its constituent elements by weathering products may differ from those observed for biotite where considerable modification of chemical composition and structure is required (Gilkes *et al.*, 1986).

The geochemical behavior of elements during weathering of a mineral is governed, among other factors, by the capacity of the alteration products to retain elements released by the parent mineral. In a broad sense, elements can be grouped as mobile, residual or intermediate on the basis of their general geochemical behavior (Loughnan, 1969). However, the structural characteristics of parent and secondary minerals may also play a role in determining the mobility of elements (Gilkes *et al.*, 1986). Several studies have shown that considerable amounts of some nominally mobile elements are retained in secondary minerals. Such apparently anomalous behavior may be attributed to the influences of the structure and chemistry of both primary and secondary minerals on the mobility of an element during weathering (Anand and Gilkes, 1984). Therefore, for a precise characterization of the geochemical behavior of an element during weathering, its mobility in different primary and secondary mineral

systems must be considered. Isovolumetric chemical weathering of mineral grains in deep horizons of lateritic profiles allows one to investigate the morphological, mineralogical and chemical changes produced solely by *in situ* chemical weathering. Alteration products in these profiles are to a large extent pseudomorphs after parent mineral grains. They have not been physically disturbed, as might occur in shallow soils, and they have not received substantial particulate additions from external sources (i.e., illuviation is minimal). Thus, a precise account can be made of the mineralogical alteration mechanisms and of the behavior of elements present in a single primary mineral grain during the weathering process. This paper describes the lateritic weathering of a chromium-bearing muscovite, and the concomitant behavior of the constituent elements.

MATERIALS AND METHODS

Altered and fresh muscovite grains were separated from a lateritic weathering profile on quartzite at the Windmill Hill railway cutting near Toodyay in Western Australia (Williams, 1975). In the upper horizons of the profile the quartzite has exfoliated and fractured, and muscovite grains are altered to various extents. In the lower horizons the muscovite grains are fresh. A block of weathered quartzite approximately $30 \times 15 \times 10$ cm³ was broken along foliation planes to expose the muscovite crystals. Grains that had experienced various degrees of weathering were picked up by tweezers under a stereomicroscope. Care was taken not to squeeze the grains between the tweezers or to otherwise physically distort them. Grains present in the single specimen block were separated on the basis of extent of weathering, as indicated by color and exfoliation.

X-ray diffraction patterns of single flakes of about 0.5 mm size were obtained with Fe-filtered CoK α radiation using a Gandolfi camera (Gandolfi, 1967). The flakes were not rotated and were positioned either parallel or normal to the beam so as to obtain diffraction patterns corresponding to these orientations. The flakes

were next rotated during exposure to the X-ray beam and a random orientation XRD pattern obtained to permit identification of mineral phases present.

Whole grains were impregnated with epoxy resin and polished thin sections made on glass slides for optical examination and quantitative elemental electron microprobe analysis (EMPA) using an ARL-SEM-Q instrument. Altered grains were also examined by scanning electron microscopy (SEM) using a Philips PSEM 505 instrument. Specimens were mounted on aluminum stubs, coated with carbon, and subsequently with gold, in a vacuum evaporator. Energy-dispersive X-ray spectral (EDXS) microanalysis was used to characterize mineral phases from their semi-quantitative chemical compositions. X-ray maps of the distribution of elements in partially weathered grains were obtained using EDAX. PV9900, an X-ray analytical software package available with the SEM. A matrix of 520×400 pixels at $81 \times$ magnification was used. A count rate of about 4000 counts/second with a count time per pixel of 300 milliseconds was used, which resulted in about 20 hours of total data collection time for each field.

Samples for TEM were prepared by two methods. Some partially weathered grains were gently crushed and fine-grained alteration products deposited onto carbon-coated copper grids. Other samples were prepared by microtomy. A fraction containing approximately 5–20- μm particles was settled into a flat-bottomed plastic tray and dried at 45°C. The dried sample was then impregnated with Spurr's resin (Spurr, 1969) and ultrathin sections cut with a diamond knife using a Reichert-Jung microtome. The ultrathin sections were floated onto water, collected on 400 mesh copper grids and coated with a thin layer of carbon in a vacuum evaporator. The ultrathin sections and the samples deposited on carbon films were investigated using a Philips 430 analytical transmission electron microscope operated at 250 kV.

RESULTS AND DISCUSSION

Mineralogy of the single flakes

XRD patterns of a single altered muscovite crystal (i.e., one flake) positioned parallel, normal and at random orientations with respect to the collimated X-ray beam are shown in Figures 1A, B and C, respectively. The flake oriented parallel to the X-ray beam gave enhanced basal reflections, and that oriented normal to the beam gave enhanced $hk0$ reflections (MacEwan, 1961). These diffraction patterns represent an advanced stage of muscovite alteration, with much kaolinite and little muscovite being present. Kaolinite is clearly the weathering product of muscovite. The basal reflections (Figure 1A) and $hk0$ reflections (Figure 1B), respectively, of kaolinite and muscovite fall on com-

mon axes demonstrating the each of the major crystallographic axes of kaolinite and muscovite are in parallel orientation. There is also a very weak ring pattern for kaolinite in Figure 1B, which indicates that although the majority of the kaolinite crystals are in parallel orientation, some kaolinite crystals, or possibly halloysite crystals, are distributed at random orientations about the c^* axis. A similar, highly-oriented growth of secondary clay minerals in weathered biotite extending from micrometer to millimeter scale was observed by Gilkes and Suddhiprakarn (1979). Oriented secondary minerals develop if the parent and secondary minerals contain similar structural units (groups, chains, sheets), and the parent mineral contains adequate amounts of elements of a residual nature (Gilkes *et al.*, 1986). Kaolinite could inherit directional information from muscovite by growing epitaxially on the surface of residual layers of muscovite, or by topotactic replacement of muscovite layers by kaolinite layers (Figure 2). During topotactic alteration, parts of the muscovite structure could be retained with little or no modification. For such an alteration to occur all Al in the tetrahedral sheets of muscovite would be removed, and one of the two tetrahedral sheets would be either removed (Mode 1) or inverted (Mode 2). The octahedral sheet of muscovite may be adopted by kaolinite with little or no modification. A consequence of this type of topotactic alteration may be that normally mobile elements (e.g., Fe, Mg) are not exposed to dissolution and are consequently retained in the secondary mineral. In this case, cations may be preserved through the inheritance of intact octahedral sheets by the kaolinite weathering product. This contrasts with mineral alterations involving complete dissolution and recrystallization where mobile elements are released from structural sites in the minerals and can be removed by diffusion and leaching. There is no apparent reason why one of the two tetrahedral sheets should be stripped preferentially from a muscovite layer in Mode 1 alteration as both sheets are crystallographically equivalent. However, it is possible that the local initiation of stripping of a tetrahedral sheet at the edge of the crystal or defect destabilizes the adjacent tetrahedra in the sheet so that they are relatively much more susceptible to dissolution than are tetrahedra in the opposed sheet.

Morphology of the single flakes

A scanning electron micrograph of a partially altered muscovite grain is shown in Figure 3. The mineral phases identified on the basis of their morphology and semiquantitative chemical composition provided by the EDXS analysis are platy kaolinite, tubular halloysite and platy muscovite crystals. Halloysite could not be distinguished from kaolinite in XRD patterns (Figure 1), probably because it is a very minor constituent and in a dehydrated state (7 Å) has an XRD pattern similar to that of kaolinite. The kaolinite crystals are

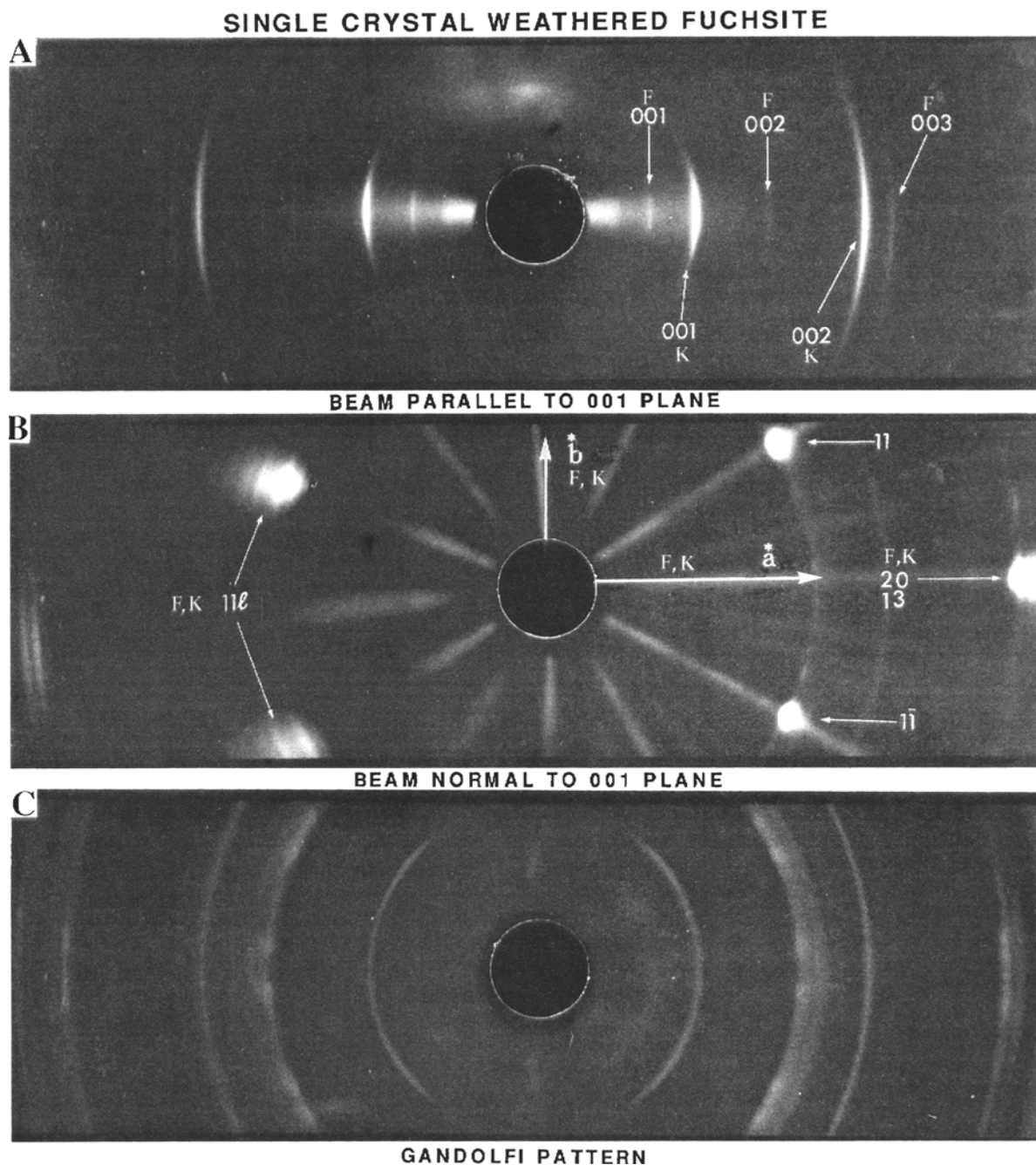


Figure 1. X-ray diffraction patterns of an altered chromian muscovite (fuchsite) flake obtained by the Gandolfi method with the X-ray beam parallel and normal to the stationary crystal; and with the crystal in random orientations. F: fuchsite, K: kaolinite.

oriented in a single direction parallel to the muscovite crystals. Due to their tubular morphology the halloysite crystals cannot conform to the three-axis parallel orientation shown by muscovite and kaolinite. The halloysite tubes are mostly parallel to the (001) plane of kaolinite and muscovite and have adopted a random orientation in this plane. This interpretation of SEM

photographs is consistent with the single-flake XRD patterns. The weak rings due to $hk0$ reflections in the single grain XRD pattern (Figure 1B) are probably mostly due to the randomly-oriented halloysite crystals, although one might also expect to observe weak basal reflections due to halloysite in this pattern (at $d = 7.2$ or 10 \AA).

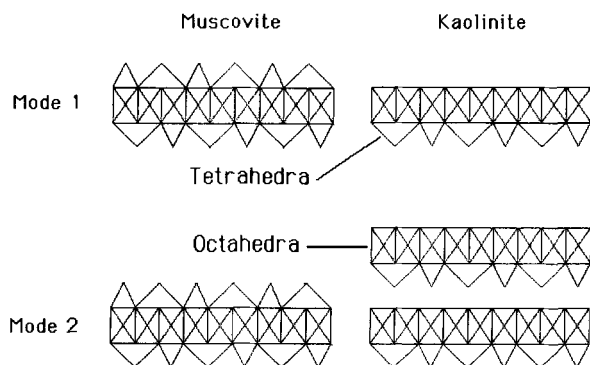


Figure 2. Two modes of topotactic transformation of chromian muscovite (fuchsite) to kaolinite by stripping of tetrahedral sheets (Mode 1) and by the insertion of octahedral sheets (Mode 2).

Weathering interface

A TEM micrograph of scrapings from the cleaved surface of an altered muscovite crystal shows that the kaolinite occurs as elongate, multiply-twinned, euhedral hexagonal crystals that are in perfectly parallel crystallographic alignment (Figure 4). These complex kaolinite assemblages form sheets that are parallel to the residual muscovite fragments, as would be anticipated for this type of epitaxial or topotaxial alteration. Kaolinite and muscovite were readily identified from their elemental compositions, which were obtained by operating the microscope with a 10 nm beam in X-ray nanoprobe mode. The selected area electron diffraction (SAED) pattern of an area shown in Figure 4 consists of a single net of hk reflections, demonstrating that parent muscovite and secondary kaolinite are in perfect parallel alignment with respect to their a and b axes. The a and b unit cell dimensions of these two minerals are too similar to be resolved by electron diffraction (i.e., XRD patterns (Figure 1), $b(\text{kaolinite}) = 8.96 \text{ \AA}$, $b(\text{muscovite}) = 8.99 \text{ \AA}$). Kaolinite crystals are elongated along their a crystallographic axis, and thus apparently grew at a faster rate in this direction.

Lattice-fringe images of sections cut perpendicular to the (001) cleavage of a partially-weathered flake show kaolinite layers exhibiting a 7 \AA spacing. These are perfectly parallel and in immediate contact with layers showing the 10 \AA spacings of muscovite (Figure 5). Similar strain-free parallel intergrowths of kaolinite with biotite (Ahn and Peacor, 1987) and wonesite (Veblen, 1983) have been reported, but it should be noted that, in contrast to muscovite, both of these micas are trioctahedral. There is no amorphous or poorly-crystalline phase separating the kaolinite and muscovite, as has been observed in some other studies of weathered mineral grains (Banfield and Eggleton, 1990). In this instance (Figure 5) alteration would appear to be advancing along the c axis, but this observation is not proof of this process as this image does not allow the

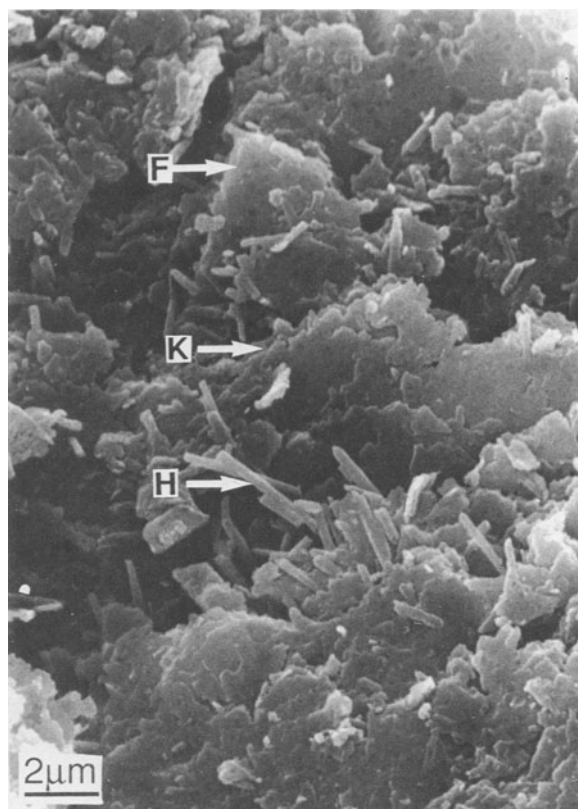


Figure 3. SEM image of a fracture surface of an altered chromian muscovite crystal. (K) oriented kaolinite plates, (H) randomly oriented halloysite crystals, (F) residual sheet of chromian muscovite.

identification of the direction of movement of the alteration front through the muscovite crystal. It is possible for a (001) interface between the two minerals to be formed by alteration proceeding along the c axis or by replacement of muscovite by kaolinite within the (001) planes. It was therefore necessary to locate all types of alteration interface to confirm the various types of alterations that may occur.

Detailed examination of several lattice-fringe images indicated that the alteration of muscovite to kaolinite may occur via either of the two modes proposed in Figure 2 that involve alteration within the (001) plane. The first mode consists of a lateral transition of one muscovite layer to one kaolinite layer resulting in a net reduction in volume (Figure 6). In the second mode, one muscovite layer transforms into two kaolinite layers resulting in a localized increase in volume (Figure 7). It appears that perfectly-aligned kaolinite develops by replacement of muscovite layers rather than by the growth of oriented kaolinite crystals on the surface of muscovite crystals. Thus, this style of alteration is most appropriately called topotactic rather than epitaxial (Banfield *et al.*, 1989). For each mode of transformation the numbers of atoms of various elements required

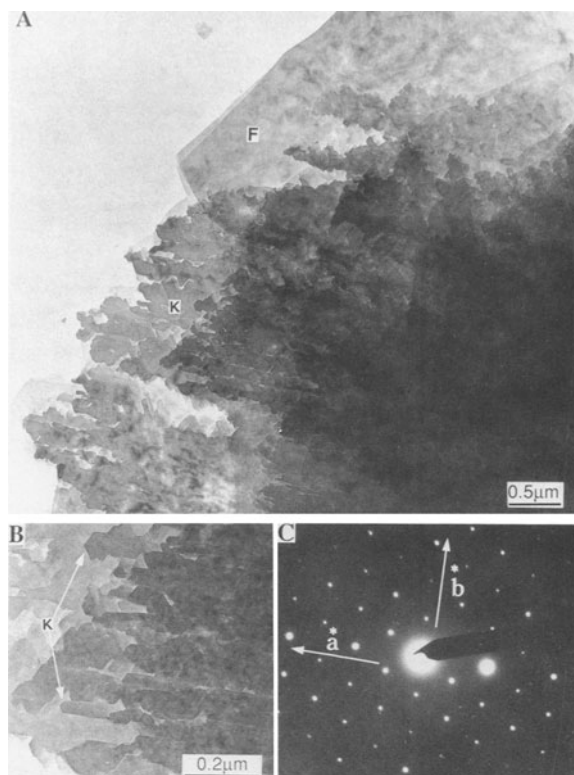


Figure 4. (A) TEM image of a partially-altered flake of chromian muscovite (F) showing oriented growth of kaolinite crystals (K). (B) A high magnification view of a part of A. (C) SAED pattern of B after correcting for its rotation relative to the image.

to be imported or removed from the reaction front for weathering of one unit cell of muscovite are shown in Table 1. For the first mode of transformation to occur some Al ions must be removed from the reaction site (Table 1), whereas for the second mechanism of transformation, additional Al is supplied from outside the reaction site. For the weathering process in a muscovite crystal to be entirely conservative of Al, Mode 1 alteration will occur about twice as frequently as Mode 2 alteration. Cations are presumably supplied to the reaction front via diffusion through solution from regions in the weathering crystal, where either complete

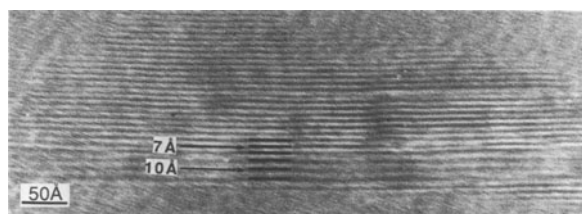


Figure 5. Lattice-fringe image of a (001) interface between chromian muscovite (10 Å) and kaolinite (7 Å), possibly due to alteration proceeding along the *c* axis direction.

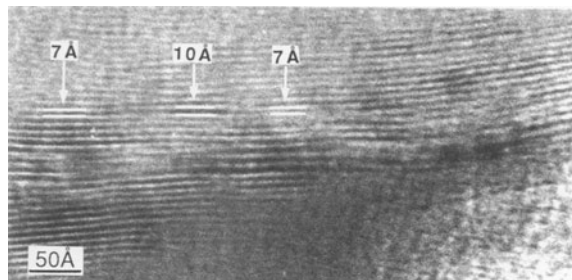


Figure 6. Lattice-fringe image showing the lateral transformation of one chromian muscovite (10 Å) layer into one kaolinite (7 Å) layer within the (001) plane.

dissolution or transformation by Mode 1 has occurred. It appears that the discontinuity in the structure at the reaction front, especially in the absence of any other structural defect, is likely to extend within the *ab* plane (which is approximately parallel to the electron beam for Figures 6, 7) as a tunnel or porous zone, and will provide a pathway for diffusion of ions to and from the reaction front. Such tunnel-like structures were observed and have been considered to transport reactant and product ions and water at interfaces for the weathering of chain silicates (Veblen and Buseck, 1980), chlorite-phlogopite (Yau *et al.*, 1984), and biotite (Ahn and Peacor, 1987).

Retention of elements

Polymers, consisting of components of octahedral and tetrahedral sheets of muscovite, may be incorporated into the kaolinite structure with little modification since the chemical composition of these sheets in muscovite is similar to that in kaolinite. A similarly extensive and equally conservative transformation of biotite to kaolinite may not be possible, inasmuch as biotite contains considerable amounts of Fe and Mg in the octahedral sheet that are not inherited by the kaolinite weathering product. Consequently, much of the Fe and Mg in biotite is mobilized to form discrete oxides (Fe), or leaves the weathering grain by diffusion (Mg) rather than being retained in the octahedral sheet

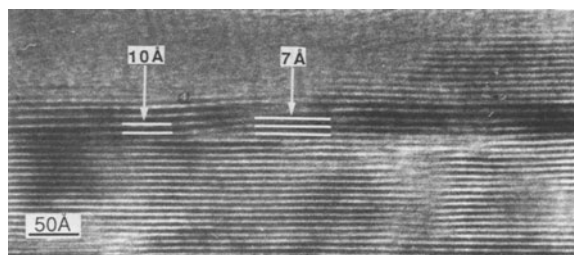


Figure 7. Lattice-fringe image showing the lateral transformation of one layer of chromian muscovite (10 Å) to two layers of kaolinite (7 Å) within the (001) plane.

Table 1. Budget of elements for the transformation of chromian muscovite (fuchsite) to kaolinite by two different modes of transformation. Values are the numbers of ions in one or two unit cells. Mode 1: one 10 Å unit cell transforms to one 7 Å unit cell of kaolinite. Mode 2: one 10 Å unit cell transforms to two 7 Å unit cells of kaolinite.

Ion	Muscovite No. of ions	Mode 1		Mode 2	
		No. of ions	Loss/gain	No. of ions	Loss/gain
Si	6.24	4.00	-2.2	8.00	1.8
Al	5.18	3.91	-1.3	7.82	2.6
K	1.71	0.00	-1.7	0.00	-1.7
Cr	0.17	0.06	-0.1	0.11	-0.1
Fe	0.11	0.02	-0.1	0.03	-0.1
Mg	0.18	0.02	-0.2	0.03	-0.1
Ti	0.16	0.00	-0.2	0.00	-0.2
O	20.00	10.00	-10.0	20.00	0.0
OH	4.00	8.00	4.0	16.00	12.0
Volume (Å ³)	474.74	332.52	-142.2	665.04	190.3
No. of cations removed			5.8		2.2
No. of cations imported			0		4.4

of kaolinite (Gilkes and Suddhiprakarn, 1979; Ahn and Peacor, 1987; Banfield and Eggleton, 1988). Thus, the octahedral sheet of biotite is probably completely dismantled. The mechanism of kaolinite crystallization from muscovite described above differs from that for muscovite as inferred by Banfield and Eggleton (1990). They reported a process involving epitactic growth of kaolinite onto an intermediate smectite phase. Their samples were taken from a weathering granodiorite containing plagioclase and K-feldspar, and which was evidently altering to different secondary minerals (e.g., smectite). The sole alteration to kaolinite/halloysite is reported here. The difference in chemical environments for the two soil profiles is probably responsible for the different weathering products.

To quantify the retention of elements during weathering of a compound muscovite-kaolinite grain, the K-content can be used as an index of the amount of unaltered muscovite in the grain. This procedure is based on the reasonable assumption that K is completely lost in the alteration of muscovite to kaolinite. Any K remaining in a kaolinized region is thus assumed to be present in residual muscovite layers. The oxides of K, Si, Al, Mg, Cr, Ti and Fe determined by EMPA, and expressed on an ignited basis for micrometer-size volumes of fresh, to almost completely altered, muscovite within a former single muscovite crystal are shown in Figure 8. When all K has been lost the SiO₂ and Al₂O₃ contents are similar to those of kaolinite (about 56% and 44%, respectively). The analyses indicate that most Si is retained and Al is wholly retained. Mg and K are lost at a constant proportion as indicated by the regression line that passes almost through the origin. Much Ti is also removed as the muscovite is altered to kaolinite, which is a surprising result as Ti is considered to be an immobile element in soils. Furthermore, Ti can substitute for Si in kaolinite (Weaver and Pollard, 1973; Anand and Gilkes, 1984). However, recent work has indicated that Ti may be rather more

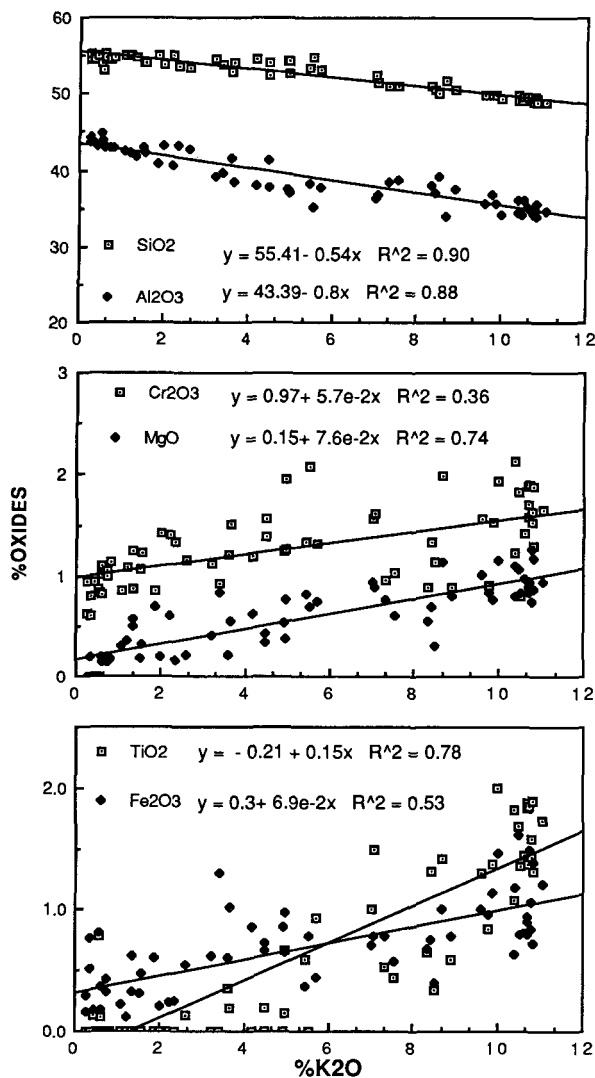


Figure 8. A plot of the concentration of oxides of Si, Al, Cr, Mg, Ti and Fe as a function of the K₂O content of micrometer-size regions within an altered chromian muscovite grain.

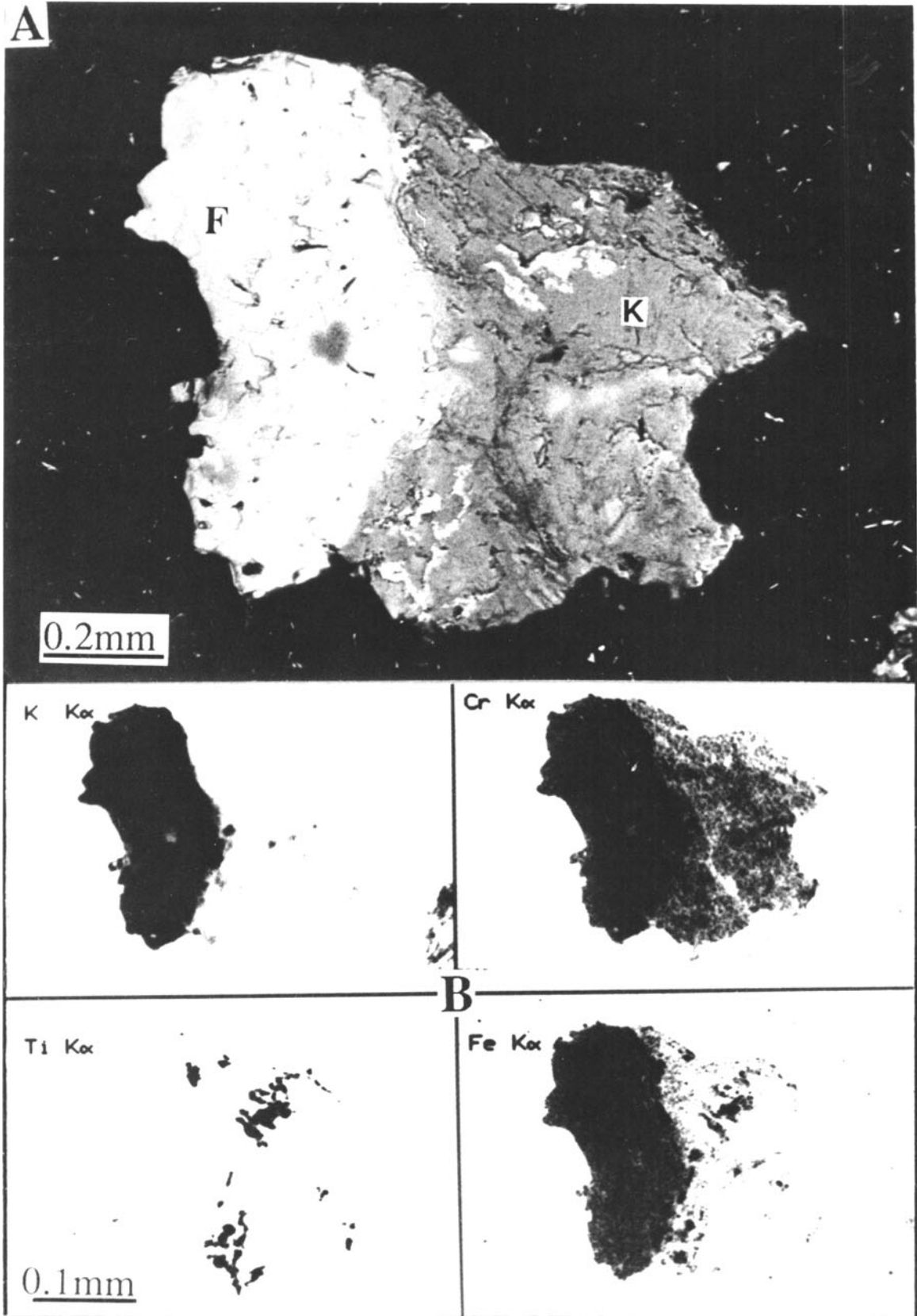


Figure 9. (A) Back-scattered electron image and (B) X-ray maps of a partially weathered chromian muscovite (fuchsite) grain. F: fuchsite, K: kaolinite.

mobile in soils than has been assumed (Milnes and Fitzpatrick, 1989). X-ray maps of the elements in muscovite show that some additional Ti is present in the mica as a discrete phase and is retained as a discrete phase in the kaolinite pseudomorphs. This phase may be rutile and/or anatase (Figure 9). The chemical changes associated with muscovite alteration to kaolinite can be described quantitatively in terms of an element budget for a single grain assuming: (1) isovolumetric weathering, and (2) Al is fully retained (Table 2). The budget shows that one unit cell of muscovite weathers to 1.37 unit cells of kaolinite with the loss of about 10% Si, 100% K, 90% Mg, 80% Fe, 50% Cr, and 100% Ti. The net change in volume is a decrease of 2.7%.

About 50% of the initial Cr and 20% of the initial Fe are retained when chromian muscovite is replaced by kaolinite. Cr and Fe commonly substitute for Al in octahedral sites in layer silicates (Matzat, 1978) so that they might be expected to be retained by kaolinite. X-ray maps of Cr and Fe demonstrate that these elements are not present as distinct phases of a size that can be detected by EMPA (Figure 9). Similarly, no discrete Cr or Fe compounds were detected by AEM (resolution ~10 nm) and are thus unlikely to be present. Their uniform distribution in the weathered kaolinite region indicates that Cr and Fe are present in the structure of kaolinite. The elemental compositions of micron-size single particles of kaolinite and halloysite determined by AEM are shown in Table 3. There is considerable variation (σ) in values obtained by this procedure due partly to: (1) the relatively unfavorable counting statistics associated with a low concentration of an element in very a small particle, and (2) the presence of small and variable amounts of residual muscovite layers within some kaolinite grains. Bearing in mind these limitations, the mean elemental composition is consistent with values obtained by microprobe analysis of completely altered zones in former

Table 2. Elemental budget for one unit cell of chromian muscovite (fuchsite) weathering to 1.37 unit cells of kaolinite based on the assumption that all Al is retained. Values are thus numbers of ions in 1 and 1.37 unit cells of muscovite and kaolinite respectively.

Ion	Muscovite	Kaolinite	% Loss/gain
Si	6.24	5.61	-10
Al	5.18	5.18	0
K	1.71	0.00	-100
Cr	0.17	0.08	-54
Fe	0.11	0.02	-78
Mg	0.18	0.02	-87
Ti	0.16	0.00	-100
O	20.00	13.70	-32
OH	4.00	10.96	174
No. unit cells	1.00	1.37	-
Volume (\AA^3)	467.70	454.9	-2.7

Table 3. Elemental composition (%) of particles of kaolinite and halloysite determined by AEM. Data are mean values for analyses of 20 particles of kaolinite and halloysite, respectively.

Element	Kaolinite		Halloysite	
	Mean	σ	Mean	σ
Al ₂ O ₃	43.24	1.78	44.59	0.90
SiO ₂	53.64	1.21	53.97	1.09
K ₂ O	0.61	0.77	0.02	0.03
TiO ₂	0.31	0.54	0.01	0.03
Cr ₂ O ₃	0.90	0.53	0.51	0.30
Fe ₂ O ₃	0.45	0.34	0.36	0.21
Total	99.14		99.46	

muscovite crystals. The mean Cr and Fe contents of halloysite are slightly lower than for kaolinite, possibly because halloysite has crystallized entirely from solution, whereas kaolinite has developed to some extent by topotactic alteration of muscovite, which would have preserved some of the Cr and Fe from the octahedral sheet of parent muscovite. Although chromium-rich kaolin-group minerals are rare they do occur in nature (Sheih and Maksimovic, 1982), being associated with hydrothermal alteration or weathering of ultramafic rocks under conditions where there is an abundance of Cr and/or a deficiency of Al. Maksimovic *et al.* (1981) described kaolinite and dickite containing 0.69% and 0.57% Cr₂O₃, respectively. Clearly, kaolin group minerals are able to accommodate structural Cr so that retention of some Cr in kaolinite formed from muscovite could be anticipated. Similarly, kaolin group minerals may accommodate substantial amounts of structural Fe (Rengasamy *et al.*, 1975; Herbillon *et al.*, 1976).

CONCLUSIONS

Two major and novel conclusions may be derived from this investigation. Firstly, the kaolinite crystals that have developed from muscovite faithfully preserved the crystallographic orientation of the parent mica. Secondly, the alteration was probably at least partly topotactic involving preservation of some components of the octahedral sheet of muscovite, which has resulted in the retention of some Cr and Fe in kaolinite. There was no evidence of the presence of amorphous intermediary compounds.

ACKNOWLEDGMENTS

We are grateful to T. M. Armitage and the technical staff of the Electron Microscopy Centre, University of Western Australia, for their assistance. B.S. gratefully acknowledges the Commonwealth Scholarship provided by the Australian International Development Aid Bureau.

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(Received 9 April 1991; accepted 12 June 1991; Ms. 2084)