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CHARACTERIZATION OF MORPHOLOGICAL FEATURES OF SOIL MICAS USING SCANNING ELECTRON MICROSCOPY

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Abstract—Morphological changes occurring in naturally weathered micas in seven residual soils from Ontario were studied. Splitting, exfoliation of the (100) planes, surface coats, filling of spaces with soil material as well as cracking and crumbling of (001) surfaces were the main morphological changes found to be taking place during natural weathering of mica particles. The significance of the observed feature in relation to the general theory of mica weathering is discussed.

Key Words—Exfoliation, Mica, Muscovite, Splitting, Weathering.

INTRODUCTION

Several reports on surface morphology of micas and micaceous vermiculites have been published (Seddoh and Robert, 1972; Jackson et al., 1973; Jackson and Sridhar, 1974; Lee et al., 1974). All the micas used in these studies were from rock samples rather than soil micas. Chemical treatments often preceded examination, a fact that makes the applications of results to natural weathering conditions of little value, since these treatments, however mild, have been shown to cause alteration of micas, particularly biotite (Protz and St. Arnaud, 1964; McKeague and Day, 1966; Arshad et al., 1972).

The purpose of this work was to characterize the stages of alteration of a variety of weathered micas from seven Ontario soils through examining the 100 and 001 surfaces, and to relate the observed features to the general theory of mica weathering.

MATERIALS AND METHODS

Mica particles were separated from seven residual soils from Peterborough and Haliburton counties, Ontario (Tarzi, 1976). The parent materials of these soils consisted of a granite, a granite-gneiss, an amphibolite, a syenite, a diorite, and a marble. The mica types and soil profile names are shown in Table 1.

Separation of mica particles

Micas were collected from several horizons of each soil profile so that the full range of features would be studied. One hundred grams of each of the selected samples were shaken in distilled water for 12 hr. The dispersed samples were wet sieved. The sand (50–2000 μm) was separated, washed with distilled water, dried at 50°C, and then fractionated by dry sieving.

Micas were separated from the 250–500 μm fraction. A hand magnet was passed through the samples to separate strongly magnetic particles. The rest of the magnetic minerals were separated from the nonmagnetic ones by a Frantz isodynamic separator model L-1 using 0.8 A current, 10° horizontal and 28° vertical tilt. The separate obtained was placed on smooth sheets of paper and gently hand shaken to allow the nonplatey minerals to roll off leaving material consisting of about 70–90% mica. These then were further purified by hand picking under a binocular microscope.

Preparation of mica for scanning electron microscopy

Eighteen to 20 particles from each sample were selected to include the full range of mica. Using a needle, these were sandwiched between two scotch-tape strips under the microscope with about half of the particle protruding out of the sandwich. Using Al stubs having adjustable slits, the sandwiches were lowered into these slits, and openings were adjusted so as to hold the sandwiches firm. The samples were coated with a 400 Å thick layer of gold-palladium (40–60%) and examined under the SEM.

A few particles (5–7) from each horizon were glued parallel to the (001) planes on Al stubs using No. 1481 silver paint and coated in the same manner as described for the examination of cleavage surfaces.

The SEM used was an ETEC Autoscan. Accelerating voltages of 5 and 20 kV were used for examining the edges and the (001) surfaces of mica flakes, respectively.

OBSERVED FEATURES

The scanning electron micrographs showed the sequence of physical changes taking place during mica weathering. The perfectly intact edges of fresh particles (Figure 1a) were rare. Splitting starts along a few planes and increases as weathering advances (Figures 1b, c) separating the layers and reaching the exfoliation stage (Figure 1d). The separating layers were occasionally

Table 1. Soils and their mica types studied.

Soil sampling name	Mica type
Snowdon marble	Phlogopite
Tweed marble	Vermiculitic phlogopite
Belmont diorite	Biotite
Amphibolite	Biotite
Catchacoma granite-gneiss	Biotite
Methuen granite	Biotite
Syenite	Biotite

bent (Figure 1c), a phenomenon speculated on by Seddoh and Robert (1972) to be tectonic deformation.

Few particles had relatively clean edges. The majority of particles were coated with material of different morphology. Irregular patchy deposits were observed on numerous particles (Figure 2a). Some particles were almost entirely coated (upper part of Figure 2b). In others, the coating material sealed the spacings leaving small areas of porous surface (lower part of Figure 2b). In some particles the coating material was deposited irregularly over the entire edge giving the latter a spongy appearance similar to the central part of Figure 2b. In others, it took the appearance of hard crust similar to carbonate deposits (Figure 2c) or sheetlike crusts covering extensively split edges (Figure 2d). Roth et al. (1966, 1968) examined coatings on micaceous vermiculites and found them to be Fe_2O_3 (80–85%) along with Al_2O_3 and SiO_2. The work of Tarzi (1976) leads us to the same conclusion.

Root-hairlike bodies were observed extending into particles as in Figure 3a from the Ah horizon of the Tweed marble soil profile. This could be a root hair or fungal hyphae. The spaces formed by splitting either remain relatively clean or are filled with mineral and possibly organic particles (Figures 3b, c, d). Perfectly smooth (001) surfaces were not found among the particles examined. The least affected ones had surfaces such as the one shown in Figure 4a. Most particles had different degrees and types of degradation (Figures 4b, c, d). Holes of different sizes and shapes appeared on the (001) surfaces of quite a few micaceous particles (Figure 5). The area around these holes were often highly affected by weathering (Figure 5b). Sharp and smooth-edged cracks that are, most probably, related to mica genesis, were rather common.

Regularly shaped crystalline particles could often be seen embedded as inclusions in the mass of the micaceous particles (Figure 5a). Protz et al. (1974) reported quartz inclusions in biotite particles from thin section studies of these soils. Upon removal these inclusions would leave regularly shaped holes with sharp boundaries (Figure 5a) that could be mistaken for fission particle tracks reported by Lee et al. (1974). The micrographs in Figures 5a and d put the claim of these workers in serious doubt. The elongated hexagonal holes on the micaceous particles they studied could very well be alien mineral sites rather than fission particle tracks. The removal of crystalline inclusions from mica particles would lead the weathering of the 001 surface as shown in Figures 4b, c, d, 5c, and d.

SIGNIFICANCE OF THE OBSERVED FEATURES

Diffusion reaction model

Some of the features observed on the electron micrographs of the mica particles suggest that the rate of replacement of interlayer K would, most probably, not fit the mathematical model for a diffusion controlled reaction generally followed in laboratory experiments of K release. The diffusion model is based on idealized conditions (MacKintosh et al., 1971) which assume: (1) that a linear relationship is maintained between $t^{1/2}$ and the distance travelled by the replacement front, (2) the displacement front moves progressively and uniformly inward from the periphery towards the center of the particle with no preferred "channels," (3) all the K displaced appears in the solution, (4) there is no inhibition due to the released K^+ in solution.

While most of these conditions may largely be met by uniform clean crack-free mica particles with evenly cut edges, they almost certainly would not be met by naturally weathered mica particles. The electron micrographs showed that the edges of these particles were very irregular. Slanted (Figure 6a), steplike (Figure 6b), and irregular (Figure 6c) edges on mica particles were common. There were numerous holes, cracks, and depressions on (001) surfaces and many were filled with finer particles.

Scott and Smith (1967) showed that particles that had cracks showed irregular weathering fronts and poorly defined color boundaries. Newman and Brown (1969) demonstrated that the nature of the crystal edge affected the rate of alteration of micas. The exchange region developed unevenly when a raggedly cut particle was used. Wells and Norrish (1968) reported that the altered edges of the mica particles used in their study were severely distorted (also shown by Boyle et al., 1967). They were not able to get positive proof as to whether the micas became simply wrinkled or whether there was also splitting and cracking. The present study provides proof that splitting, separation of layers, and cracks were not only present, but were the most striking morphological changes observed. Scott and Smith (1967) pointed out that there should be a rapid release of K by the exposure of interlayer surfaces and that this must be considered along in the kinetics of interlayer K. Wells and Norrish (1968) reasoned that if splits and cracks were present, the treatment solution might be able to penetrate the mica and cause increased reaction rates. It could do so by mass flow along the wrinkles,

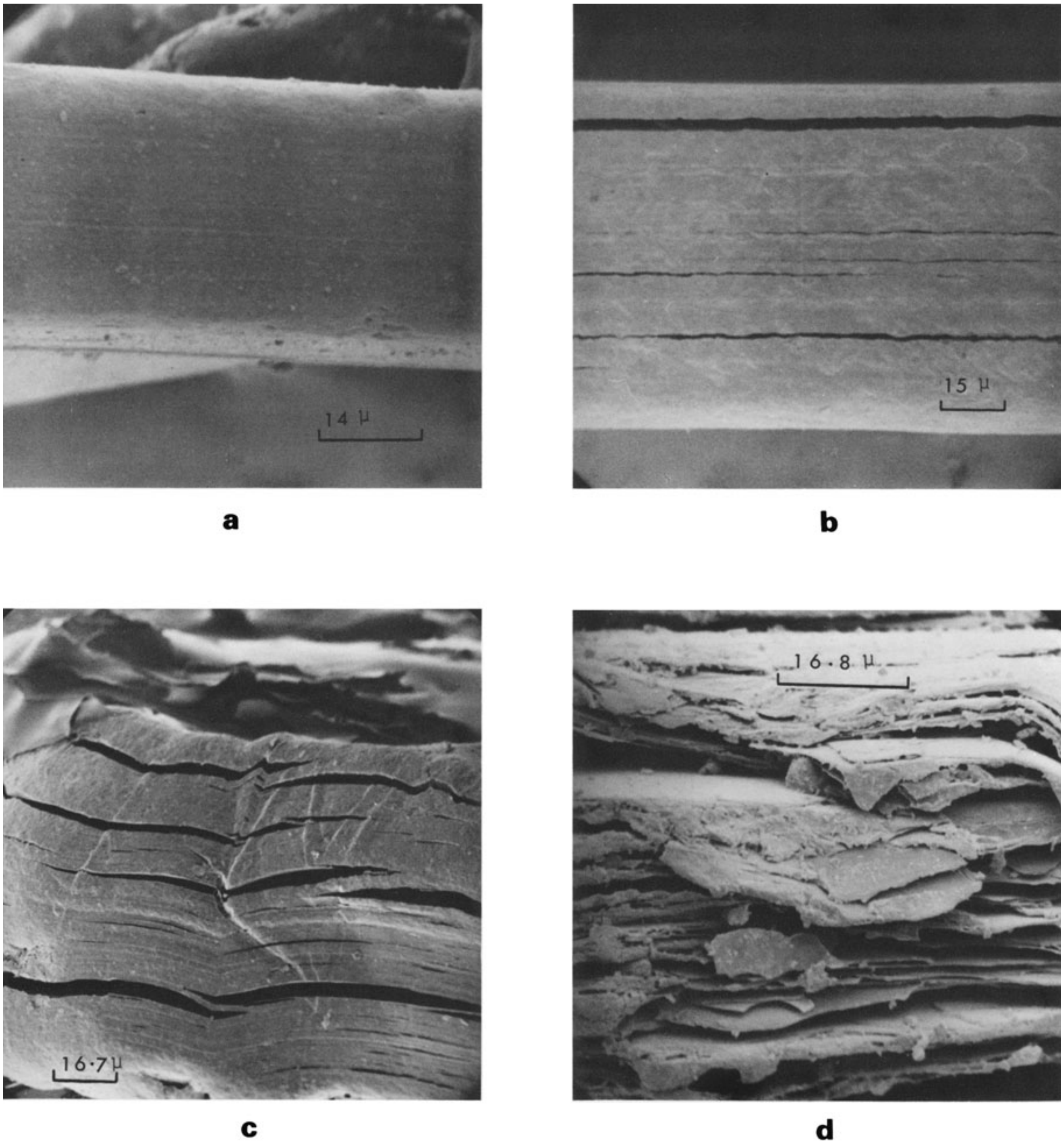

Fig. 1. Types of (100) surface splitting found in various mica particles: a—an intact particle; b—a particle with initial splitting; c—a particle with greater splitting and tectonic deformation; d—a particle with major exfoliation.

or by direct entry through a split. As the split developed, it could have the effect of constantly carrying with it the starting site for diffusion of a replacing cation into the layer lattice structure and likewise shortening of the path for the escaping K.

Rausell-Colom et al. (1965) maintained that the slow rate of natural weathering of mica was due to the presence of K^+ in solution causing a reduction in the rate of diffusion. Evidence supporting this proposal has been discussed by Tarzi and Protz (submitted for publication).

The amorphous coatings discussed earlier can also slow down diffusion and cation exchange reactions. Evidence to this effect has already been presented by

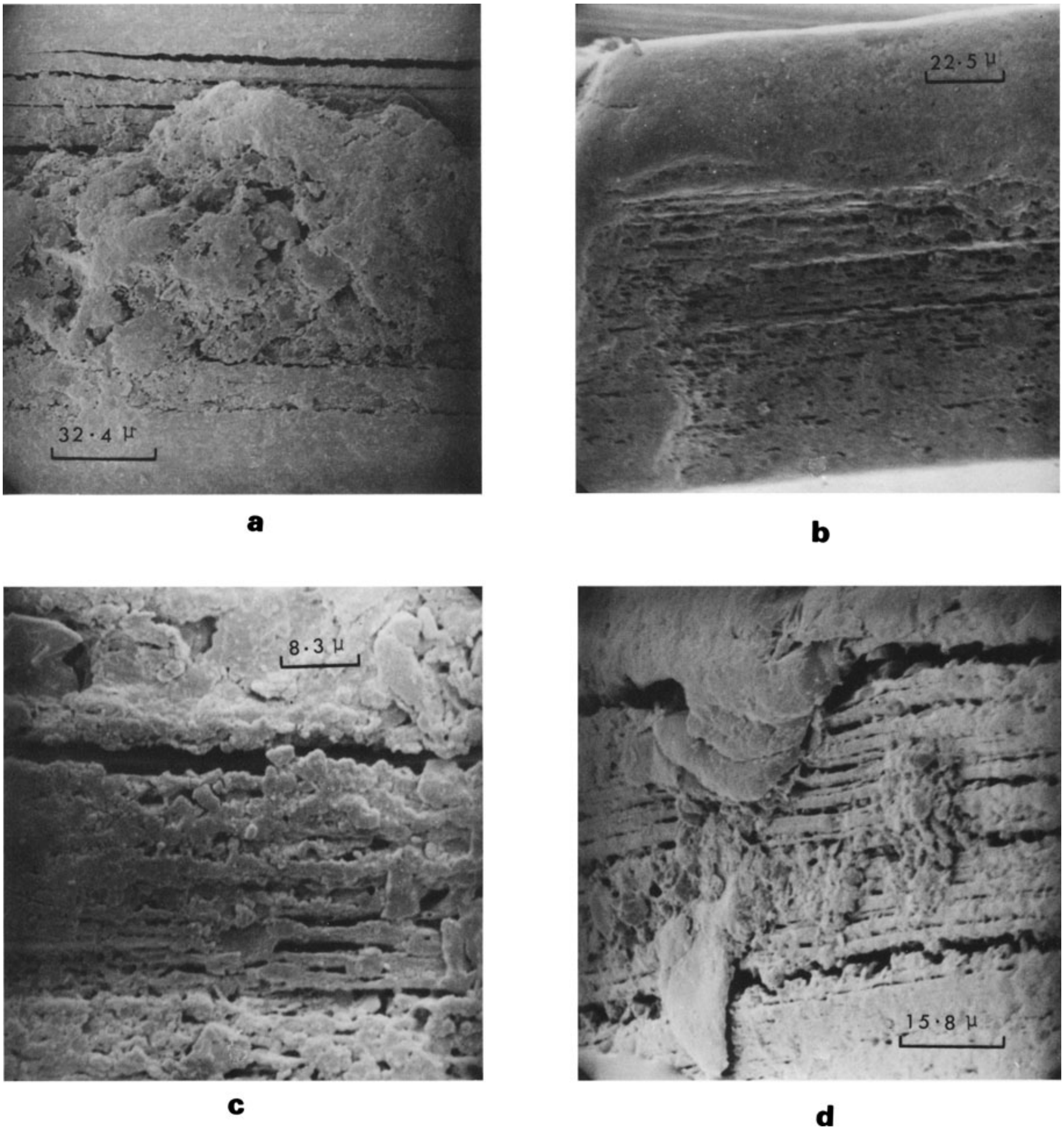

Fig. 2. Types of (100) surface coats found on various mica particles: a—relatively thick crust on a mica particle from the Tweed marble soil profile; b—completely coated (upper part), spongy appearance (middle part) slightly porous coating (lower part) on a mica particle from Snowdon marble soil profile; c—crusty deposit on a mica particle from the Snowdon marble soil profile; d—sheetlike crusts on mica from Tweed marble soil profile.

Roth et al. (1968). The coats can also undergo irreversible dehydration, yielding hard cementing material that can hold the layers of the micaceous particles together preventing mechanical separation.

Filling material

The filling material observed in the spaces can affect the course and intensity of weathering. In addition different mineral particles are sources of cations. The spaces can form sites for biological activity. Material of organic origin (possible root hairs or fungal hyphae), if present, could induce mechanical as well as chemical weathering.

Layer vs. edge weathering

Scott and Smith (1967) showed by laboratory studies of K release that both layer and edge weathering take place during K displacement from macro-mica (flakes

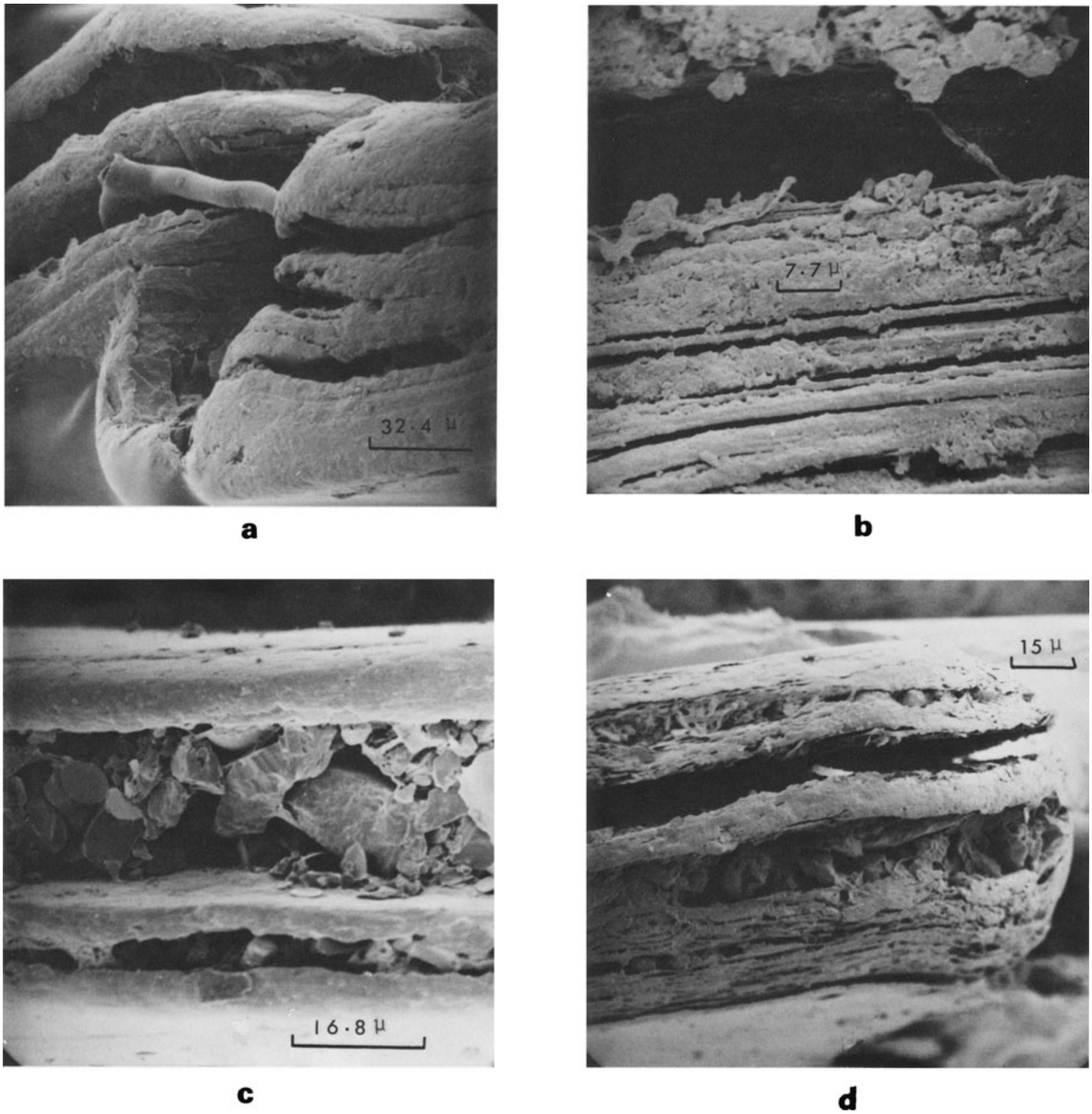

Fig. 3. Various types of materials in interlayer voids of mica particles: a—root hair extending into mica particles from the Tweed marble soil; profile; b—filamentous material in particle from the Methuen granite soil profile; c—mineral particles of various shapes and sizes in a particle from the Catchacoma granite-gneiss soil profile; d—mineral particles and small mica weathering products in a particle from the Methuen granite soil profile.

(10 to 25 mm). The electron micrographs presented in this paper indicate that layer rather than edge weathering was dominant in natural weathering, despite the fact that the particle size of the micas examined was in the range of 250–500 μm. Differences in thickness between the central part and peripheral zone of the particles and characteristics of edge weathering were not apparent. Splitting, wide separation of layers and exfoliation attributed by Scott and Smith (1967) to layer weathering, were, as pointed out previously, major morphological features of the mica particles in the present study. The splitting of particles, according to Scott and Smith (1967) was brought about by the expansion force from the initial exchange of K^+ at the edge of the particles. The release of this stress imposed by expansion may possibly be effected more easily by a complete

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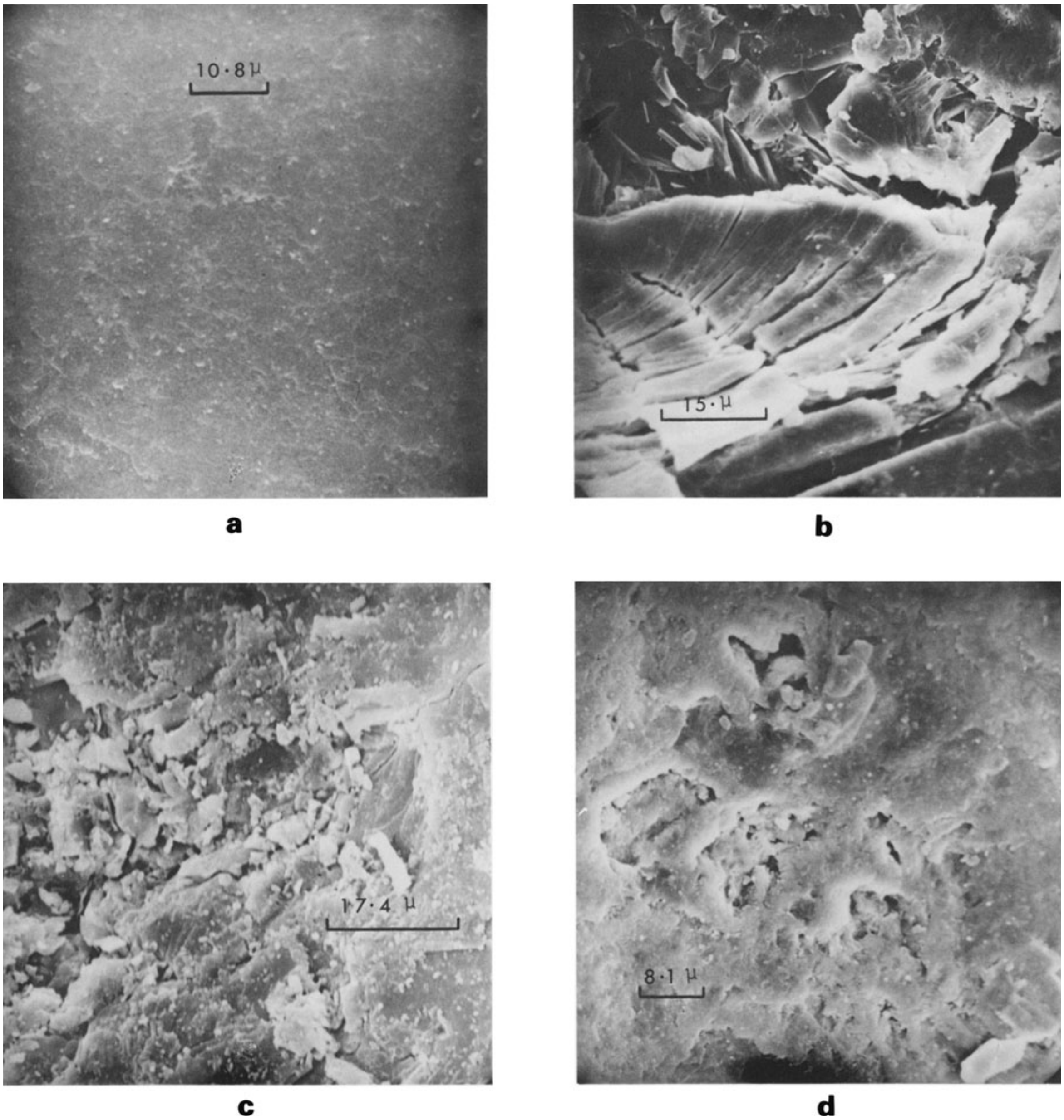

Fig. 4. Types of weathering features found on the (001) surfaces of mica particles: a—intact mica showing initial cracks and thin coats from the Snowdon marble soil profile; b—highly weathered sides of a hole on a mica particle from the Tweed marble soil profile; c—weathered particles on a mica from the Catchacoma granite-gneiss soil profile; d—holes and crusts on mica from the Methuen granite soil profile.

separation of layers than by a uniform marginal expansion (Reichenbach and Rich, 1969). Similarly, Rausell-Colom et al. (1965) suggested that as natural weathering was slow, the replacement of K was not so orderly, and once a particular interlayer region had opened up, continued replacement in that region would be favored over the opening up of other interlayer regions. Therefore, interstratification would be expected during natural weathering. Similar suggestions were made by Scott (1968), Rich (1968), and Newman and Brown (1969), all of which are supported by the common occurrence of interstratified phases in the micas of this study, and the morphological features discussed above.

The irregularity of particle edges, a prominent feature of particles examined, has also been reported to favor layer weathering leading to interstratified phases.

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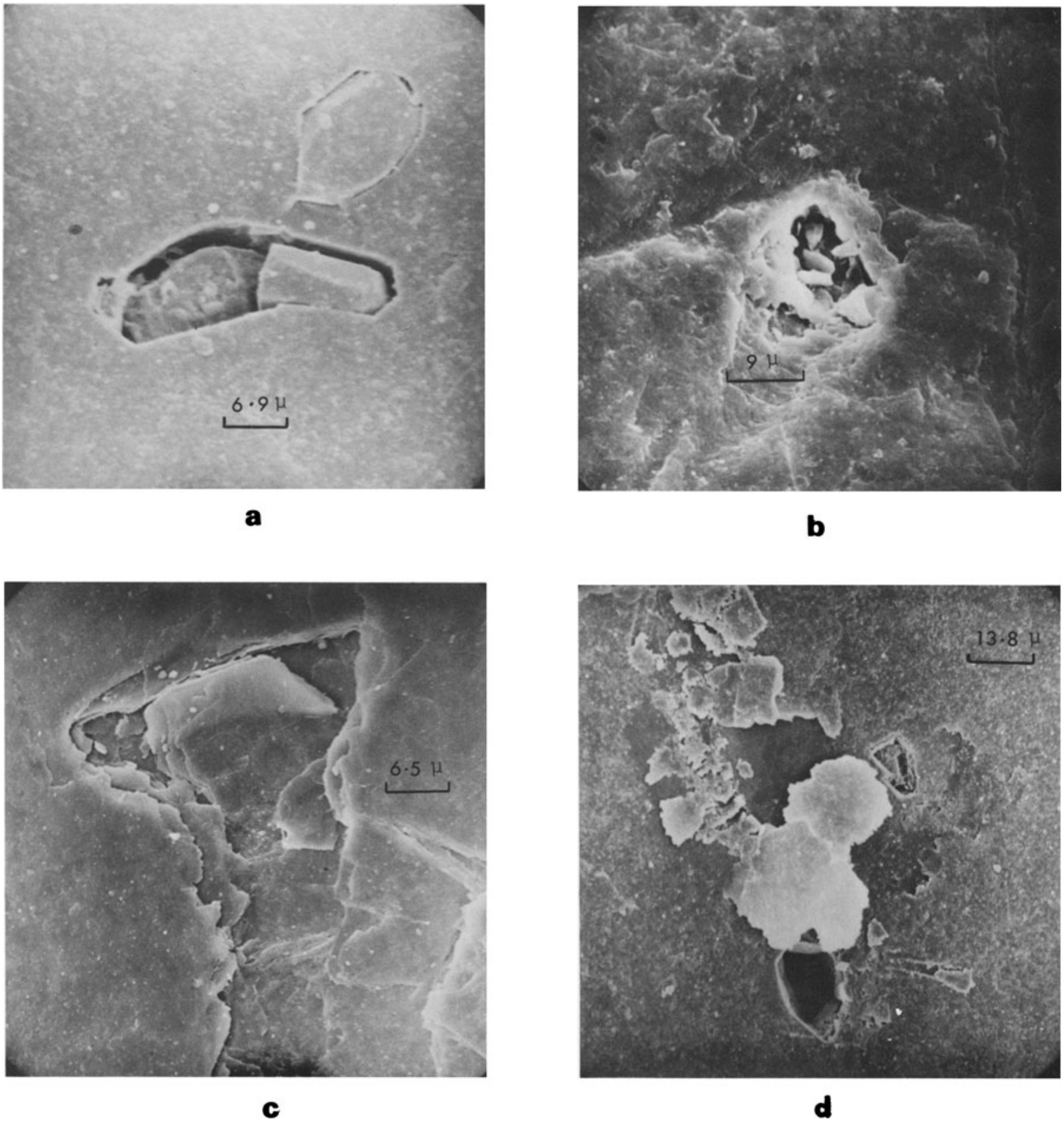
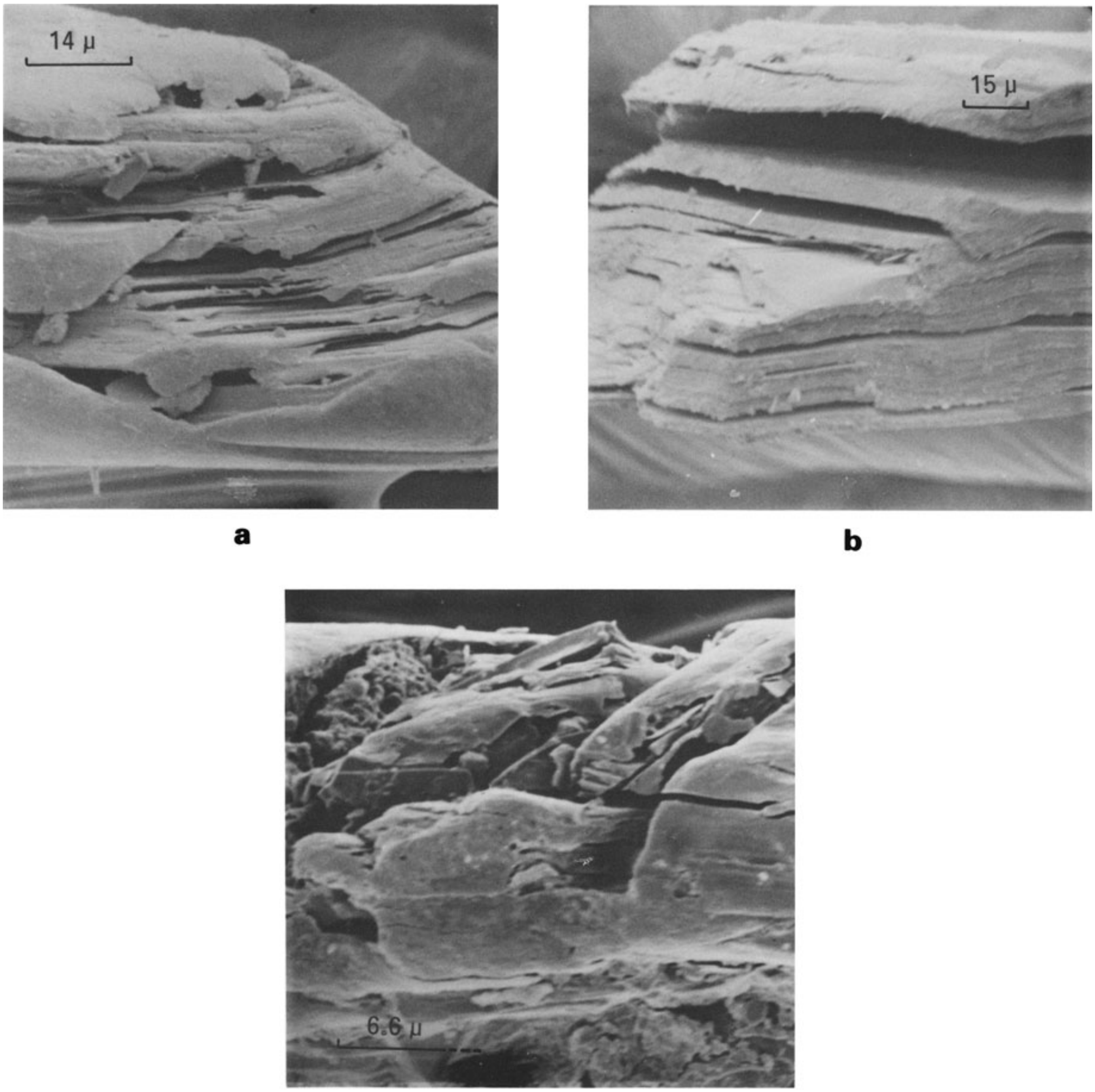


Fig. 5. Crystalline mineral inclusions and modes of weathering found on (001) surfaces of various mica particles: a—included minerals in mica from the syenite soil profile; b—hole being refilled with weathered particles, and crack on right hand side on a mica from the Snowdon marble soil profile; c—extent of weathering around hole on a particle from the syenite soil profile; d—holes and altered platelets on a particle from the syenite soil profile.

In an irregular edge, the initiation of exchange reaction would be at different points relative to the center creating a nonuniform exchange front. This means that exchange from some layers would occur at a more rapid rate than from others resulting in interstratification (layer weathering). LeRoux and Rich (1969), using X-ray diffraction analysis, ultramicrotome sections and other microscopic studies, showed that the exchange of Mg by Rb and Sr, did not progress in a uniform front. Electron and light microscope investigations indicated that the edges of these micas are steplike. These observations along with the findings of the present study indicate that layer weathering and interstratification are important in natural mica weathering even for the coarser particles. This casts some doubt on the validity of the proposed layer versus edge weathering as a



a

b

c

Fig. 6. Morphology of edges on mica particles: a—slanted edges on a particle from the Belmont diorite soil profile; b—step-like edges on a particle from the syenite soil profile; c—irregular edges on a particle from the Catchacoma granite-gneiss soil profile.

mechanism responsible for the observed particle size effect on K release. Many studies on particle size effect on K release have been carried out on ground-up samples (Reichenbach and Rich, 1969; LeRoux and Rich, 1969; MacKintosh et al., 1971) which have been shown to have irregularly edged particles resulting from grinding (LeRoux and Rich, 1969). Yet, the finer fractions have been shown to release less K than the coarser ones.

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REFERENCES

Arshad, M. A., St. Arnaud, R. J. and Huang, P. M. (1972) Dissolution of trioctahedral layer silicates by ammonium oxalate, sodium dithionite-citrate-bicarbonate, and potassium pyrophosphate: *Can. J. Soil Sci.* **25**, 19–25.

Boyle, J. R., Voigt, G. K. and Sawhney, B. L. (1967) Biotite flakes: alteration by chemical and biological treatment: *Science* **155,** 193–195.

Jackson, M. L., Lee, S. Y., Brown, J. L., Sachs, I. B. and Syers, J. K. (1973) Scanning electron microscopy of hydrous metal oxide crusts intercalated in naturally weathered micaceous vermiculite: *Soil Sci. Soc. Am. Proc.* **37,** 127–131.

Jackson, M. L. and Sridhar, K. (1974) Scanning electron microscope and X-ray diffraction study of natural weathering of phlogopite through vermiculite to saponite: *Soil Sci. Soc. Am. Proc.* **38,** 843–847.

Lee, S. Y., Jackson, M. L. and Sachs, I. B. (1974) Fission particle tracks in micas and micaceous vermiculites: *Soil Sci. Soc. Am. Proc.* **38,** 423–428.

LeRoux, J. and Rich, C. I. (1969) Ion selectivity of micas as influenced by degree of potassium depletion: *Soil Sci. Soc. Am. Proc.* **33,** 684–690.

MacKintosh, E. E., Lewis, D. G. and Greenland, D. J. (1971) Dodecylammonium-mica complexes—1. Factors affecting exchange reactions: *Clays & Clay Minerals* **19,** 209–218.

McKeague, J. A. and Day, J. H. (1966) Dithionite- and oxalate-extractable Fe and Al as aids in differentiating various classes of soils: *Can. J. Soil Sci.* **46,** 13–22.

Newman, A. C. D. and Brown, G. (1969) Delayed exchange of potassium from some edges of mica flakes: *Nature* **223,** 175–176.

Protz, R. and St. Arnaud, R. J. (1964) The evaluation of four pretreatments used in particle-size distribution analyses: *Can. J. Soil Sci.* **44,** 345–351.

Protz, R., Gillespie, J. E. and Brewer, R. (1974) Micromorphology and genesis of four soils derived from different rocks in Peterborough County, Ontario, Canada: *Soil Microscopy, Proc. 4th Int. Working Meeting on Soil Micromorphology,* Kingston, Ontario, pp. 481–497.

Rausell-Colom, J. A., Sweatman, T. R., Wells, C. B. and Norrish, K. (1965) Studies in the artificial weathering of mica: *Experimental Pedology, Proc. Univ. Nottingham 11th Easter Sch. Agric. Sci.,* pp. 40–72.

Reichenbach, H. Graf von and Rich, C. I. (1969) Potassium release from moscovite as influenced by particle size: *Clays & Clay Minerals* **17,** 23–29.

Rich, C. I. (1968) Mineralogy of soil potassium. In *The Role of Potassium in Agriculture* (Edited by Kilmer, V. J., Founts, S. E. and Brady, N. C.), pp. 79–108. Am. Soc. Agron., Crop Sci. Am., Soil Sci. Soc. Am.

Roth, C. B., Jackson, M. L., De Villiers, J. M. and Volk, V. V. (1966) Surface colloids on micaceous vermiculite: *Trans. Comm. II & IV, Int. Soc. Soil Sci.,* Aberdeen, pp. 217–221.

Roth, C. B., Jackson, M. L., Lotse, E. G. and Syers, J. K. (1968) Ferrous-ferric ratio and CEC changes on deferration of weathered micaceous vermiculite: *Isr. J. Chem.* **6,** 261–273.

Scott, A. D. (1968) Effect of particle size on interlayer potassium from soils and micaceous minerals with solutions containing sodium tetraphenylboron: II. Biotite: *Soil Sci. Soc. Am. Proc.* **26,** 41–48.

Scott, A. D. and Smith, S. J. (1967) Visible changes in macro mica particles that occur with potassium depletion: *Clays & Clay Minerals, Proc. 15th Nat. Conf.* pp. 69–81.

Seddoh, F. K. and Robert, M. (1972) Intérêt de l'utilisation due microscope électronique à balayage pour l'étude des micas et de leur évolution (évolution expérimentale et dans le milieu naturel): *Bull. Soc. Fr. Mineral. Cristallogr.* **95,** 75–83.

Tarzi, J. G. (1976) Weathering of mica minerals in selected Ontario soils: Unpublished Ph.D. Thesis, Land Resource Science, University of Guelph, Guelph, Ontario.

Tarzi, J. G. and Protz, R. (1978) Increased selectivity of naturally weathered biotites for potassium: *Soil Sci. Soc. Am. Proc.* (submitted for publication).

Wells, C. B. and Norrish, K. (1968) Accelerated rates of release of interlayer potassium from micas. *Trans. 9th Int. Congr. Soil Sci.* Adelaide, Australia, **2,** 683–694

Резюме- Изучались морфологические изменения,наблюдающиеся в естественно выветренных слюдах в семи образцах элювия,отобранных в Онтарио. Было обнаружено,что расщепление,расслаивание плоскостей/100/,поверхностные покровы,заполняющие промежутки почвенным материалом,также как растрескивание и смятие поверхностей/001/ были главными морфологическими изменениями,происшедшими в период естественного выветривания частиц слюды. Обсуждается значение выявленных особенностей для общей теории выветривания слюды.

Kurzreferat- Morphologische Veränderungen,welche in natürlich verwetterten Glimmern in sieben Eluvialböden in Ontario vorkommen,wurden untersucht.Spaltung,Abblätterung der (100) Ebenen,Oberflächendecken,das Füllen von Plätzen mit Erdmaterial wie auch Brechen und Zerbröckeln von (001) Oberflächen waren die hauptsächlichen morphologischen Veränderungen,welche während der natürlichen Verwetterung der Glimmerteilchen Platz nehmen.Die Bedeutung der beobachteten Eigenschaften im Vergleich zu der allgemeinen Theorie der Glimmerteilchen wird diskutiert.

Résumé-Les changements morphologiques survenant dans des micas naturellement altérés à l'air dans sept sols résiduels d'Ontario ont été étudiés. La fendaison et l'exfoliation des plans(100),l'apparition de couches sur les surfaces,le remplissage d'espace de matière provenant du sol,ainsi que le craquement et l'émiettement des surfaces(001) étaient les principaux changements morphologiques survenant pendant l'altération naturelle des particules de mica à l'air.La signification de cette observation est discutée en relation avec la théorie générale de l'altération du mica.

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