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FORMATION OF AN INTERSTRATIFIED MINERAL BY EXTRACTION OF POTASSIUM FROM MICA WITH SODIUM TETRAPHENYLBORON

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Abstract—Interlayer K was extracted with sodium tetraphenylboron solution from a powdered sericite heated to the temperature of dehydroxylation and quenched to room temperature. By this procedure, sericite of the $2M_1$ type was changed to an interstratified mineral. The X-ray diffraction properties of this interstratified mineral are similar to "rectorite".

INTRODUCTION

INTERSTRATIFIED or mixed-layer minerals were reported many years ago by many investigators. But only a few data on the synthesis of mixed-layer minerals have been reported. Ueda and Sudo (1966) synthesized an interstratified mineral from sericite under hydrothermal conditions. Tomita and Sudo (1968*a* and *b*) synthesized an interstratified mineral from pre-heated sericite ($2M_1$ type) by acid treatment at room pressure and Tomita and Sudo (1971) formed an interstratified mineral from sericite by treatment with $LiNO_3$.

Mica weathering in soils has been attributed to a loss of K and a gain in water. Loss of K changes micas into expanded layer silicates (Jackson and Sherman, 1953). Artificial extraction of interlayer K from mica in the laboratory at one atmosphere have been carried out with aqueous salt solutions (Barshad, 1948, 1954; Mortland, 1958; Rausell-Colom *et al.*, 1965) and laboratory weathering of the micaceous mineral to mixtures of vermiculite and regularly and randomly interstratified mica–vermiculite was reported by Rich and Cook (1963). Extraction of interlayer K in muscovite by treating the mineral with molten $LiNO_3$ at 300°C was carried out by White (1956, 1958). The application of sodium tetraphenylboron (NaTPB) to the extraction of interlayer K from micaceous minerals was initiated by Hanway (1956), De Mumbrum (1959, 1963), Scott, Hunziker and Hanway (1960), Scott and Reed (1962*a* and *b*). Potassium depletion of micas has produced more interstratification when the particles are smaller (Scott, 1968), but formation of regular type of mixed-layer minerals in the process of the extraction of K from micaceous minerals using NaTPB have not been reported The present writers succeeded in forming an interstratified mineral from sericite using NaTPB. As shown in the previous study by Tomita and Sudo (1968*a* and *b*), an interstratified mineral was formed from pre-heated sericite at one atmosphere. This is an easy procedure and its result seems to be pertinent to the discussion of the origin of interstratified clay minerals.

EXPERIMENTAL

Sericite from the Goto mine, Nagasaki Prefecture, Japan was used as a starting material. Fractions less than 2μ were collected by the sedimentation method. The $< 2\mu$ material was dried in air and was used for experiments. The sample was investigated and confirmed as a sericite of the $2M_1$ type polytype and did not contain any interstratified minerals. The X-ray powder pattern of the original sample is shown in Fig. 1. The differential thermal analysis curve is shown in Fig. 5. The dried sample was heated at 800°C for one hour. After heating the specimen was cooled and placed in an aqueous NaTPB solution and kept at room temperature for various periods of time. After the desired period, the sample was washed with distilled water by filtering several times. The sample, dried in air, was investigated by X-ray, chemical, differential thermal and i.r. absorption analysis. The procedure used at the beginning of this study for treating the heated sample with the NaTPB reagent was as follows: a 0·35g sample of the heated sericite was put in 70 ml of solution in which 3·5g of NaTPB was included. The potassium determinations were made with a flame photometer.

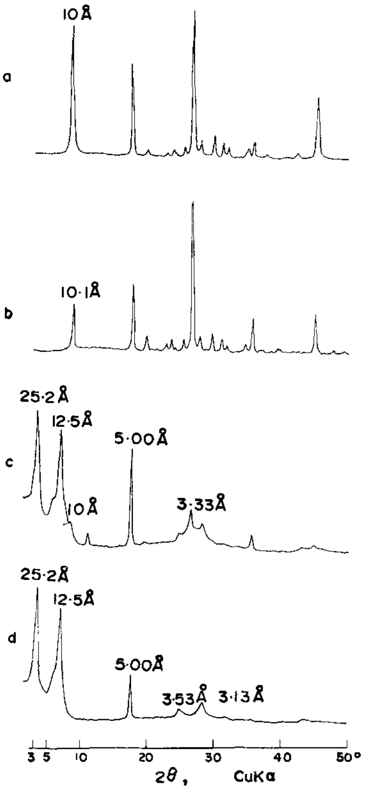

Fig. 1. X-ray diffraction patterns of unheated sericite, heated sericite and specimen treated with NaTPB. *a*, unheated sericite; *b*, sericite heated to 800°C for 1 hr; *c*, specimen altered from heated sericite by treatment with NaTPB for 3 hr; *d*, specimen altered from the heated sericite by treatment with NaTPB for 114 hr.

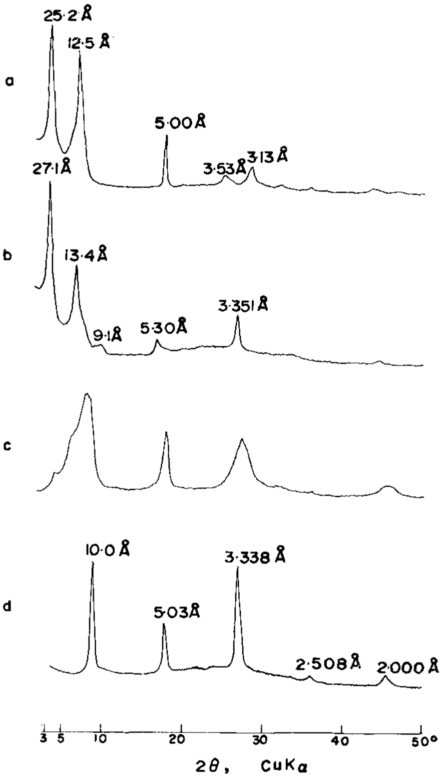

Fig. 2. X-ray diffraction patterns for the altered specimen after various treatments. *a*, specimen altered from heated sericite by treatment with NaTPB for 114 hr; *b*, treated with ethylene glycol; *c*, heated to 200°C for 1 hr; *d*, heated to 600°C for 1 hr.

RESULTS

X-ray diffraction analysis

Oriented specimens of the NaTPB treated samples were prepared and changes in basal spacing of the regular and ethylene glycolated samples were determined by X-ray diffraction. The effect of time of reaction of 0·35g of heated sericite in 70 ml of NaTPB solution (3·5g of NaTPB) is shown in Fig. 1.

A specimen formed after treatment for 3 hr was an interstratified mineral, but contained a small amount of sericite. After a 114 hr reaction time the sample showed strong 25·2 and 12·5Å peaks. For small NaTPB/sample ratios an interstratified structure was not formed even after long reaction times. Because the 114 hr reaction product was the most homogeneous interstratified mineral, it was selected and its properties were investigated.

The X-ray diffractogram of this specimen gave a rational series of reflections corresponding to a *d*(001) of 25·2 Å (Fig. 2a). Ethylene glycol solvation caused the 25·2Å reflection to shift to 27·1Å, which was accompanied by a rational sequence of 00*l* reflections indicating complete expansion of the clay (Fig. 2b). After heating to 200°C for 1 hr a broad 10 Å peak appeared indicating incomplete dehydration (Fig. 2c). After heating to 600°C specimen yielded a rational series of reflections based on 10Å. The X-ray powder data for the altered sericite are listed in Table 1. The powder data and the behaviour of the basal reflection upon treatment with ethylene glycol and heat show that this specimen is nearly a regularly interstratified mineral of mica and hydrous mica having two layers of water molecules with certain amounts of cations between silicate layers. The properties of the altered specimen are similar to "rectorite" (Bradley, 1950) or "allevardite" (Brindley, 1956).

A Fourier transform method after MacEwan (1956a) was used to deduce the nature of interstratification of the interstratified mineral. The

Table 1. X-ray powder data for the unheated sericite and the altered specimen after various treatments, $\lambda(\mathrm{Cu}K_\alpha) = 1{\cdot}5418$ Å

	A			*B*		*C*		*D*	
hkl	*d*(Å)	*I*	*hkl*	*d*(Å)	*I*	*d*(Å)	*I*	*d*(Å)	*I*
002	10·0	84	001	25·2	100·0	27·1	100·0	10·0	90·0
004	5·01	37	002	12·5	71·5	13·4	86·7	5·03	40·2
110	4·47	12	003			9·1	10·1	3·338	100·0
$11\bar{1}$			004					2·508	7·2
111	4·29	3	005	5·00	10·1	5·30	12·6	2·000	14·3
022	4·11	4	007	3·53	4·6				
112	3·95	2	008	3·13	6·7	3·351	22·2		
$11\bar{3}$	3·90	6	0010	2·50	0·9				
023	3·74	9	0012	2·06	2·1				
$11\bar{4}$	3·49	9							
024 006	3·34	100							
114	3·20	11							
$11\bar{5}$	3·12	1							
025	2·99	16							
115	2·868	11							
$11\bar{6}$	2.797	8							
$13\bar{1}$ 200	2·585	9							
$20\bar{2}$ 131	2·560	16							
008	2·508	5							

A: unheated sericite; *B*: altered sericite; *C*: treated with ethylene glycol; *D*: heated at 600°C for 1 hr.

equation employed in this calculation as formulated by MacEwan (1956*a*) can be written

$$W_{(R)} = \sum_R \frac{I}{E|F|^2} \cos 2\pi\mu_R R$$

where E, $|F|^2$, μ_R, are values at the position of the intensity maximum and I is the integrated intensity. μ_R is the reciprocal spacing, and the $|F|^2$ values of dioctahedral mica type layer with $1\mathrm{K}^+$, $1\mathrm{H_2O}$ in interlayers were used for the Fourier transform. Such a structure is considered to be proper for the interstratified mineral formed in this experiment. The combined Lorentz and polarization factors function used was $(1+\cos^2 2\theta/\sin 2\theta)$. Figure 3 shows the result of the Fourier transform of the basal reflection of the specimen. It was calculated using seven reflections. In the figure, *A* represents a mica layer and *B* represents a hydrous mica layer. The outstanding peaks of types *AB* and *ABAB* indicate a marked tendency for alternation of two different layers. Calculated peak heights given above the curve are for $P_A = 0{\cdot}55$, $P_B = 0{\cdot}45$, $P_{AA} = 0{\cdot}24$, $P_{AB} = 0{\cdot}76$, $P_{BA} = 0{\cdot}93$, $P_{BB} = 0{\cdot}07$, where *A* is mica layer and *B* is hydrous mica layer. Result of Fourier transform for the interstratified mineral is plotted as (*F*) in Fig. 4. The figure is based on a graph proposed by Sato (1965). In the figure, $P_{AA} = \alpha, P_{AB} = 1-\alpha, P_{BB} = \beta, P_{BA} = 1-\beta, \beta = \mathrm{K}\alpha + (1-\mathrm{K})$, $\mathrm{K} = P_A/P_B$ were used. Using this equation all the interstratified structures consisting of two kinds of layers can be plotted on the graph shown in Fig. 4. The random structures are plotted on the diagonal dotted line and regular type of mixed-layer

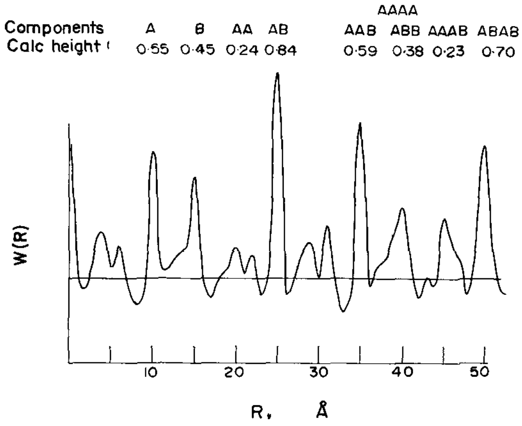

Fig. 3. Fourier transform of basal reflections (seven reflections) of the interstratified mineral (114 hr reaction product).

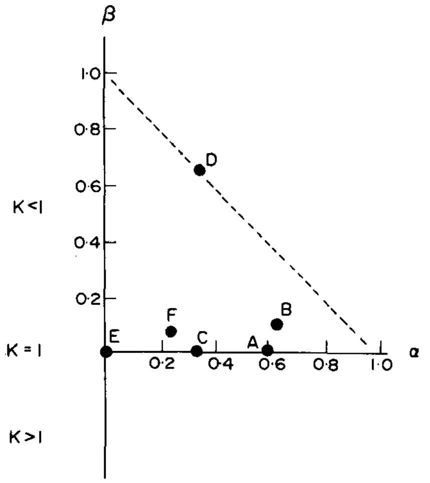

Fig. 4. Result of Fourier transform for the altered mineral (*F*) and some examples of illite–montmorillonite interstratified minerals examined. (*A*) and (*B*) are the specimens from Kamisunagawa, and (*C*) is from Honami mine. (*D*) is the specimen investigated by MacEwan (1956*b*). (*E*) is the 1:1 regular structures reported by Sudo *et al.* (1962); Brindley (1956), and Tomita and Sudo (1968*b*), where $P_{AA} = \alpha$, $P_{BB} = \beta$, and $K = P_A/P_B$.

minerals are distributed along the axes of coordinates. (*A*) and (*B*) are the specimen from Kamisunagawa in Hokkaido, Japan, which were investigated by Kobayashi and Oinuma (1960), and (*C*) is from Honami mine in Nagano Prefecture, Japan, reported by Sudo *et al.* (1962). (*D*) is the specimen investigated by MacEwan (1956*b*) using the Fourier transform method. (*E*) is the 1:1 regular structures reported by other investigators (Sudo *et al.*, 1962); Brindley, (1956); Tomita and Sudo, 1968*b*).

Differential thermal analysis

A differential thermal analysis curve of the interstratified mineral is shown in Fig. 5 together with those of the unheated sericite and sericite heated at 800°C for 1 hr. The endothermic peak at about 680°C in the curve of the unheated sericite sample is not shown in the heated sericite. The NaTPB-treated sample shows a lower temperature (620°C) endothermic reaction. This peak is considered attributable to the dehydroxylation of structural water in the alumina-silicate layer. The first endothermic peak at 123°C in the curve of the altered specimen is attributable to dehydration of adsorbed water and interlayer water, and the second endothermic peak was considered to be due to dehydration of cation associated water in the interlayers.

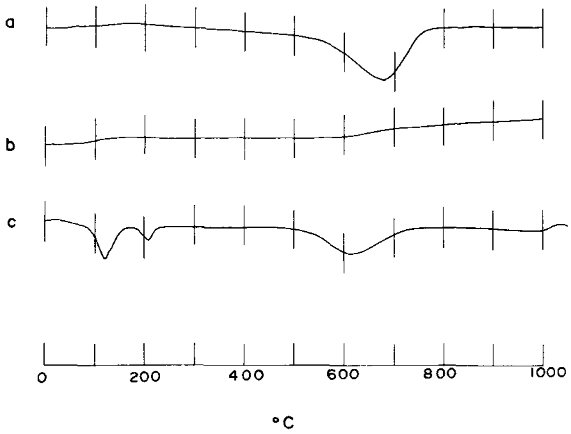

Fig. 5. Differential thermal analysis curves of heated, unheated sericite and altered sericite. *a*, unheated sericite; *b*, sericite heated to 800°C for 1 hr; *c*, specimen altered from the heated sericite by treatment with NaTPB.

I.R. absorption spectra

As shown in Fig. 6a, the unheated sericite had bands at 3640, 1020, 920, 825 and 800 cm^{-1}. The band at 3640 cm^{-1} is caused by the O–H stretching variation and the band at 920 cm^{-1} is assigned to the O–H–Al^{3+} vibration (Stubicǎn and Roy, 1961*a* and *b*). I.R. absorption spectra of the interstratified mineral formed from pre-heated sericite by treatment with NaTPB is shown in Fig. 6c together with those of the unheated sericite and the sericite (Fig. 6b) heated at 800°C for 1 hr. In the curve of the heated sample, absorption bands related to the hydroxyl groups disappeared. This agrees with the experimental results of Serratosa (1960), Grim and Kulbicki (1961) and Heller *et al.* (1962). The interstratified mineral showed absorptions bands at about 3640 and 1640 cm^{-1}. The 1640 cm^{-1} band is due to adsorbed water vibration. In addition to these bands, a broad adsorption band at 3400 cm^{-1} is observed, which is due to the adsorbed water in interlayers and is considered to be the same kind as that observed in absorption spectra of montmorillonite and hydrated halloysite.

Chemical analysis

Chemical analysis data for the interstratified mineral and the unheated sericite are listed in Table 2. The chemical data of the two samples are very similar, but the data show that the unheated sericite has a higher content of K_2O and lower content of $H_2O(-)$ as compared with the altered specimen. The value of $H_2O(+)$ of the interstratified mineral is a little large for a mica-like structure. The data of $H_2O(-)$ was obtained below

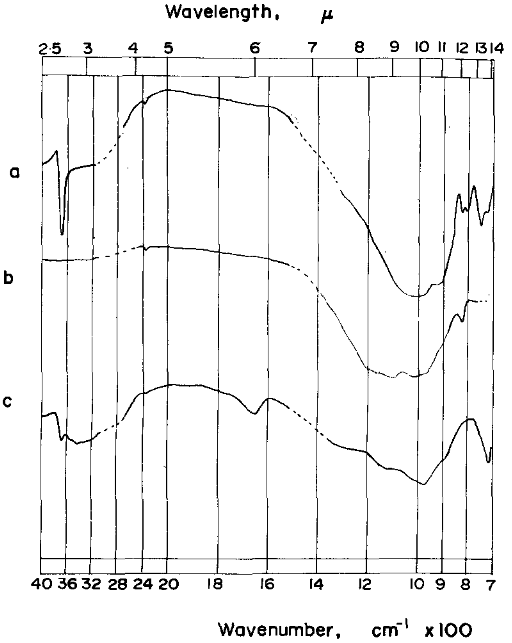

Fig. 6. I.R. absorption spectra of unheated, heated sericite and altered sericite. *a*, unheated sericite; *b*, sericite heated to 800°C for 1 hr; *c*, specimen altered from the heated sericite by treatment with NaTPB.

Table 2. Chemical compositions of the unheated sericite and the altered sericite

	(*a*)	(*b*)
SiO_2	47·24%	47·02%
TiO_2	0·38	0·13
Al_2O_3	35·04	34·70
Fe_2O_3 FeO	0·59	0·40
MnO	tr.	tr.
MgO	0·21	0·20
CaO	0·16	0·12
K_2O	8·75	3·60
Na_2O	1·37	0·80
$H_2O(+)$	5·52	8·01
$H_2O(-)$	0·32	4·70
Total	99·58%	99·68%

(*a*) unheated sericite (Analyst: K. Tomita).

(*b*) altered sericite (Analyst: M. Dozono).

110°C, and it is thought that the adsorbed water in interlayers could not be completely expelled at this temperature. Some amounts of $H_2O(+)$ should be counted as $H_2O(-)$. These facts indicate that some of the potassium ions between silicate layers were removed by treatment with NaTPB and hydration in interlayers has occurred. The structural formula of the unheated sericite is

$$\begin{pmatrix}\mathrm{Ca}\ 0{\cdot}01\\ \mathrm{K}\ \ 0{\cdot}73\\ \mathrm{Na}\ 0{\cdot}17\end{pmatrix}\begin{pmatrix}\mathrm{Al}\ \ 1{\cdot}83\\ \mathrm{Fe}^{3+}\ 0{\cdot}03\\ \mathrm{Mg}\ \ 0{\cdot}02\end{pmatrix}\begin{pmatrix}\mathrm{Si}\ 3{\cdot}10\\ \mathrm{Al}\ 0{\cdot}88\\ \mathrm{Ti}\ 0{\cdot}02\end{pmatrix}\mathrm{O}_{9{\cdot}58}(\mathrm{OH})_{2{\cdot}42}$$

and the NaTPB-treated sample gives the following structural formula

$$\begin{pmatrix}\mathrm{Ca}\ 0{\cdot}01\\ \mathrm{K}\ \ 0{\cdot}32\\ \mathrm{Na}\ 0{\cdot}10\end{pmatrix}$$

$$\begin{pmatrix}\mathrm{Al}\ \ 2{\cdot}07\\ \mathrm{Fe}^{3+}\ 0{\cdot}02\\ \mathrm{Mg}\ \ 0{\cdot}02\end{pmatrix}\begin{pmatrix}\mathrm{Si}\ 3{\cdot}24\\ \mathrm{Al}\ 0{\cdot}75\\ \mathrm{Ti}\ 0{\cdot}01\end{pmatrix}\mathrm{O}_{10{\cdot}00}(\mathrm{OH})_{2{\cdot}00}\mathrm{H}_2\mathrm{O}_{1{\cdot}92}.$$

DISCUSSION

Data on the synthesis of mixed-layer minerals are considered to be important for the interpretation of the mechanism of formation of regular mixed-layer minerals. Only a few syntheses of interstratified minerals have been reported. Iiyama and Roy (1963) synthesized an interstratified mineral from pure chemical reagents under hydrothermal condition. Ueda and Sudo (1956) succeeded in forming a regular type of mixed-layer mineral from mica, aluminium sulphate and magnesium carbonate under hydrothermal conditions (250°–300°C, 60–120 atm). Tomita and Sudo (1968*a* and *b*) succeeded in forming an interstratified structure from pre-heated mica by acid treatment at room pressure. The present authors succeeded in forming a regular type of mixed-layer mineral from pre-heated $2M_1$ sericite by treatment with NaTPB at room temperature and pressure. The interstratified mineral showed the same properties as rectorite (Brown and Weir, 1963). In our experiments, it was found that sericite should be heated up to the dehydroxylation temperature in order to change it into an interstratified structure. Tomita and Sudo (1968*b*) used a flake of muscovite ($2M_1$ type) for an experiment to investigate the structural change after heating to dehydroxylation temperature range. A Laue photograph irradiated perpendicular to a cleavage plane of heated mica flake at 800°C showed radial streaks, whereas that of the unheated specimen showed clear hexagonal spots. The radial streaks suggest some stacking disorder in the c^* direction. Existence of stacking disorder in heated mica means that some potassium ions in interlayers of heated mica were leached out by the chemical treatment more easily than those in unheated mica. Formation of a regularly interstratified structure was considered to be due to alternate leaching of potassium ions from heated mica by treatment with NaTPB. The present results suggest an ordered alternation of layer charge distribution in heated mica ($2M_1$ type). Polarity of layer charge distribution in mica has been predicted by Sudo, Hayashi and Shimoda (1962). In most montmorillonite minerals, random interstratification resulted from treatment with ethylene glycol as indicated by Green-Kelly (1955), and Tettenhorst and Johns (1963). This was suggested to be due to random distribution of layer charge.

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Résumé—Le potassium interfeuillet a été extrait, par une solution de tétraphénylborate de sodium, d'une séricite en poudre chauffée à la température de déshydroxylation et ramenée à la température ambiante. Par cette méthode, la séricite du type $2M_1$ a été transformée en un minéral interstratifié. Les propriétés de ce minéral interstratifié en diffraction des rayons X sont semblables à celles de la "rectorite".

Kurzreferat—Zwischenschicht-K wurde mittels Natrium Tetraphenyl boronlösung aus einem Serizitpulver, das auf die Dehydroxylierungstemperatur erhitzt und dann auf Zimmertemperatur abgeschreckt worden war, extrahiert. Durch diesen Vorgang wurde Serizit des $2M_1$ Typs in ein zwischengeschichtetes Mineral verwandelt. Die Röntgenbeugungseigenschaften dieses zwischengeschichteten Minerals sind ähnlich denen von "Rectorit".

Резюме — Из размолотого серицита нагретого до температуры дегидроксилизации и охлажденного до комнатной температуры раствором тетрафенилборного натрия экстрагировался промежуточный слой K. Этой процедурой серицит типа $2M_1$ был преобразован в слоистый минерал. Рентгенографическое исследование показало, что диффракционная картина этого слоистого минерала такая же как у ректорита.