AN ANALYSIS OF X-RAY DIFFRACTION LINE PROFILES OF MICROCRYSTALLINE MUSCOVITES*

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Abstract — Previous studies of the line profiles of the basal reflections of microcrystalline muscovites were refined by an adaptation of the method developed by Maire and Méring. In order to evaluate the variation of interlayer spacings, the method required only relative values of Fourier coefficients, without the correction for instrumental broadening, which was the source of one of the most critical problems previously. Instead of $K\alpha$ radiation, $K\beta$ radiation was used to record line profiles since difficulties associated with the separation of $K\alpha_1$ and $K\alpha_2$ radiations could not be overcome satisfactorily.

The data reconfirmed that the line broadening of 00l reflections was due not only to a small particle-size effect, but also to structural disorders involving the variation of the interlayer spacings. For the four specimens investigated here, the mean squares of the variation of interlayer spacings ranged from 0 to 0.0358, the square roots of which were inversely proportional to the total number of interlayer cations. It is considered that the observed distortions were mainly attributed to non-uniform interlayer spaces between silicate layers arising from an irregular distribution of interlayer cations. The data also indicated that the crystallites of all four specimens consisted of a similar number of layers. The method showed promise for the study of the nature and extent of structural disorders in micas or other silicate minerals.

INTRODUCTION

THE 00l DIFFRACTION line profiles of microcrystalline muscovites were analyzed (Kodama, 1965) to obtain information concerning the structural imperfection involving variable interlayer spacings. The present paper improves and extends that study.

As compared with the ideal composition of muscovite, the chemical composition of the microcrystalline muscovites may be characterized by a slightly high content of Si and by a slightly low total population of interlayer cations, indicating a very limited amount of solid solution of the type, $KAl \rightarrow Si$. Besides this, there was no appreciable excess of structural water and no detectable expanding or hydrated layers. Considering that a certain range of compositional variations is also observable among macroscopic muscovites, the limited solid solution may not be significant for specifying the minerals. Consequently, the very fine size of the microcrystalline muscovites would seem to be the most significant difference from the macroscopic muscovites.

In spite of their mutual resemblance and their close similarity to *ideal* muscovite, the microcrystalline muscovites give a considerable variety of diffraction broadening of the basal reflections. The earlier work aimed at interpreting the broadening revealed that the line broadening was due not only to a small particle-size effect, but also to crystal defects as deduced from the deviation from the properties of an ideal muscovite-layer (Kodama, 1965).

Although the previous results were obtained by the application of the Fourier transform followed in the well-established Houska–Warren method (1954), the following factors may have introduced significant errors: (1) the use of a muscovite flake as a standard for the instrumental line broadening correction, when the specimens studied were all powder samples, (2) the effect of unseparated $K\alpha_1$ and $K\alpha_2$ radiations on line profiles, (3) the unavoidable "umbrella effect" in the low angle region where the first order basal reflection appears, (4) the direct effect of the error due to the normalization of the Fourier coefficients on the estimation of the distortion. These factors have been reconsidered, a refinement has been undertaken and the results obtained are presented herein.

In their study on the graphitization of carbon, Maire and Méring (1958, also Maire, 1967) pro-

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posed an improved method for analyzing the modulation function $G_{hk}(Z)$, which will be defined later. It represents a modification of the Houska–Warren method (1954) with which the previous study (Kodama, 1965) was done. The Maire–Méring modification, adapted to layer silicates in the present investigation has the advantage that it can be handled rather simply and yet the potential errors can be avoided or diminished.

For the sake of clarity, the derivations of the equations for the diffraction theory are repeated briefly and reorganized here to fit the analysis of clay minerals (layer silicates) in general.

THEORETICAL TREATMENT

Consider one crystallite consisting of N parallel layers of muscovite whose translation vectors are **a**, **b** in the layer, and **c** normal to the layers.

The position of the origin of the unit cell $m_1 m_2$ of layer m_3 is given by

$$\mathbf{R}_m = m_1\mathbf{a} + m_2\mathbf{b} + m_3\mathbf{c} + \Delta(m_1\, m_2\, m_3)$$

where $\Delta(m_1\, m_2\, m_3)$ is the displacement of the unit cell $m_1\, m_2\, m_3$ that allows for distortions within the crystallite. Let **s** be the diffraction vector defined $\mathbf{s} = (\mathbf{S} - \mathbf{S_0})/\lambda$ where $\mathbf{S_0}$ and $\mathbf{S}$ are unit vectors in the directions of the incident and diffracted beams, respectively. Since the structure of a given single layer corresponds to its Fourier transform $\phi(\mathbf{s})$ the intensity in electron units for one crystallite consisting of N layers is given by

$$I_{eu} = \phi_{(s)}\phi_{(s)}{}^* \sum_m \sum_{m'} \exp\left[(2\pi i s)(\mathbf{R}_m - \mathbf{R}_{m'})\right].$$

$$(1)$$

The intensity observed is the sum of the averages of the intensities diffracted from each of a large number of crystallites. Assuming that the interference between crystallites is incoherent and that the distortions are too small to change the structure factors significantly for all unit cells, the average intensity for each layer is then given by

$$\frac{\bar{I}_{eu}}{N} = \frac{\phi^2}{N} \sum_m \sum_{m'} \overline{\exp\left[(2\pi i s)(\mathbf{R}_m - \mathbf{R}_{m'})\right]}. \qquad (2)$$

By letting $\mathbf{r}_n = \mathbf{R}_m - \mathbf{R}_{m'}$ where $n = |m - m'|$ and by grouping the double sum of equation (2) into two terms of $n = 0$ and $n \neq 0$, equation (2) can then be arranged in the following form;

$$\frac{\bar{I}_{eu}}{N} = \frac{\phi^2}{N}\left[N + 2\sum_{n=1}^{N-1}(N-n)\overline{\exp(2\pi i s r_n)}\right].$$

The general expression of the modulation function $G_{hk}(Z)$ along the reciprocal line (hk) is therefore

given by

$$G_{hk}(Z) = \frac{\bar{I}_{eu}}{N\phi^2} = 1 + 2Re \sum_{n=1}^{N-1}\left(\frac{N-n}{N}\right)\overline{\exp(2\pi i s r_n)}$$

$$(3)$$

where Re means the real part of the complex quantity. For $(00l)$ reflections, the scalar product sr_n can be replaced by Zz_n where Z is a continuous variable along the reciprocal line (00) and z_n is the interlayer spacing between an arbitrary layer and its nth neighbour. Since the number of layers (N) generally varies from one crystallite to another, the weight fraction, α_N, of crystallites consisting of N layers is introduced, then the modulation function for the basal reflections can be expressed by

$$G_{00}(Z) = 1 + 2Re \sum_N \alpha_N \sum_{n=1}^{N-1}\left(\frac{N-n}{N}\right)\overline{\exp(2\pi i Z z_n)}.$$

$$(4)$$

If there is no correlation in the succession of elementary spacing the average of the product is equal to the product of the averages (Méring, 1949).

$$\overline{\exp(2\pi i Z z_n)} = \overline{\exp(2\pi i Z z_1)^n} \qquad (5)$$

Let δ be the difference between any elementary spacing and the mean spacing d_{001} between two successive layers. Then:

$$z_1 = \bar{d}_{001} + \delta$$

and one point that is located on the reciprocal line can be determined using the following expression with any integer l_0.

$$Z = \frac{l_0}{d_{001}} + t.$$

Thus $\exp(2\pi i Z z_1)$ of equation (5) expands to $\exp(2\pi i l_0) \times \exp(2\pi i l_0\delta/\bar{d}_{001}) \exp(2\pi i \bar{d}_{001} \cdot t) \exp(2\pi i t\delta)$. Since the quantities t and δ are both small, $\exp(2\pi i t) \approx 1$, and $\exp(2\pi i l_0) = 1$, then equation (5) becomes

$$\overline{\exp(2\pi i Z z_1)^n} = \overline{[\exp(2\pi i l_0\delta/d_{001})]^n}$$
$$\times \exp(2\pi i n \cdot \bar{d}_{001} \cdot t).$$

Since δ is small, the exponential will be approximated equal to the first three terms of its expanding power series.

$$\exp(2\pi i l_0\delta/\overline{d_{001}}) = 1 + 2\pi i l_0\delta/\overline{d_{001}} - 2\pi^2 l_0\delta^2/\overline{d_{001}}{}^2.$$

By definition, the value of $\bar{\delta}$ is zero, so that

$$\overline{\exp(2\pi i l_0 \delta / \overline{d_{001}})} = 1 - 2\pi^2 l_0^2 \overline{\delta^2} / \overline{d_{001}}^2.$$

Equation (4) then becomes

$$G_{00}(Z) = G_{00l_0}(t) = 1 + 2Re \sum_N \alpha_N \sum_{n=1}^{n=N-1} \frac{N-n}{N}$$
$$\times \left(1 - 2\pi^2 l_0^2 \frac{\overline{\delta^2}}{\overline{d_{001}}^2} \cdot\right)^n$$
$$\times \exp(2\pi in \overline{d_{001}} \cdot t) \qquad (6)$$

Equation (6) indicates that the modulation function $G_{00}(Z)$ can be expressed by two separate terms, one of which includes distortions and another which does not include distortions. It is conceded that there is no correlation between crystallite size and distortion, equation (6) can be replaced by

$$G_{00l_0}(t) = 1 + 2Re \sum_{n=1}^{n=\infty} H_n \left(1 - 2\pi^2 l_0^2 \frac{\overline{\delta^2}}{\overline{d_{001}}^2}\right)^n$$
$$\times \exp(2\pi in \overline{d_{001}} \cdot t) \qquad (7)$$

where $H_n = \sum_{N=n}^{N=\infty} \alpha_N [(N-n)/N]$ for a given value of n, which is equivalent to the probability that the nth layer with respect to an arbitrary layer is found in any one crystallite. The equation (7) indicates that the profile $G_{00l_0}(t)$ of each basal reflection $(00l_0)$ is represented by the Fourier series, whose coefficients of the nth order are

$$A_n(00l_n) = H_n \left(1 - 2\pi^2 l_0^2 \frac{\overline{\delta^2}}{\overline{d_{001}}^2}\right)^n. \qquad (8)$$

Equation (8) is similar to that derived by Houska and Warren.

Consider the difference between the Naperian logarithms of the Fourier coefficients for two successive reflections.

$$\ln A_n(00l_0) - \ln A_n(00, l_0+1) = n2\pi^2 \frac{\overline{\delta^2}}{\overline{d_{001}}^2}(2l_0+1).$$
$$(9)$$

In equation (9), if the difference of the two logarithms changes linearly with n, the hypothesis that the succession of elementary spacings is independent of the succession of neighbouring spacings is justified. Hence if the linear relationship is established, $\overline{\delta^2}$ could be calculated from the slopes of the straight lines.

Remark 1

Since the Houska–Warren method involves plotting $\ln A_n(00l_0)$ against l_0^2 for two successive basal reflections and extrapolating the plots to $l_0 = 0$ for $\ln H_n$ the error due to normalization of the Fourier coefficients directly affects the results. The major advantage of the present method is that it is based on the difference between the Fourier coefficients for two successive basal reflections and consequently the normalization error does not change the tangents of the straight lines.

Remark 2

Although it is expected theoretically that the straight line determined by equation (9) should intercept the origin, many practical cases show that the lines do not pass exactly through the origin. This is because the intensity measurements at the base of a diffraction peak are never sufficiently precise to provide the Fourier coefficients free from errors due to the normalization procedure. As stated in Remark 1, however, the method depends on only the relative values of the coefficients, so that the errors do not affect the slopes of the lines and the values are not affected.

Remark 3

Using the same arguments as in Remark 2, the present method eliminates practically the effect of the instrumental broadening on the determination of $\overline{\delta^2}$. In the previous study a muscovite flake was used as a standard for the instrumental broadening correction. Because the samples investigated are powder the use of a muscovite flake as a standard would introduce a certain error. Since it is almost impossible to obtain a powder sample of macrocrystalline muscovite which gives neither strain or particle-size effects on its line profiles, the present method which avoids the correction is attractive.

Remark 4

The term, H_n, concerning particle-size can be deduced from equation (8) after the value $\overline{\delta^2}$ is once determined. If the variation of H_n is represented as a function of n, the form of H_n near the origin would be approximately $H_n = 1 - n/\overline{N}$ is the average number of layers in a crystallite. Thus, in the range where n is small, H_n should vary linearly with n. The inverse of the slope of this linear portion would give $\overline{N}$. However, it should be noted that, contrary to the determination of $\overline{\delta^2}$, values of H_n would be affected by the instrumental broadening.

Summary of analytical procedure

(i) To establish line profiles $G_{00l_0}(t)$ of two successive basal reflections with the best precision available;

(ii) To check that the line profiles are symmetrical;

(iii) To normalize the two line profiles by equal-

izing the area of each of the reflections to unity;

(iv) To perform the Fourier transform of $G_{00l_0}(t)$ in order to obtain the Fourier coefficients $A_n(00l_0)$ and $A_n(00, l_0 + 1)$;

(v) To plot $\ln A_n(00l_0) - \ln A_n(00, l_0 + 1)$ against n and to calculate $\overline{\delta^2}$ from the slope;

(vi) To plot H_n against n according to equation (8) and to evaluate the average number of layers $\bar{N}$ from the slope of the linear portion.

EXPERIMENTAL

The specimens for study

For the purpose of the present study, four micro-crystalline muscovite samples of hydrothermal origin were selected on the basis of their purity and of the different degrees of line broadening. Two of these samples had previously been investigated and two others were newly added. The samples were separated by sedimentation into the $-20\,\mu$ fraction. X-ray examinations indicated the absence of any heterophase crystalline impurity and no detectable expanding layer as a component in the fractionated specimens. The types of polymorphs, mean interlayer spacings and b-dimensions were determined from the X-ray diffraction data. The results are all listed in Table 1 with the chemical data.

In order to obtain the best diffraction profiles of basal reflections, well-oriented specimens on glass slides were carefully prepared.

X-ray diffraction technique

Regardless of the type of polymorphism of the microcrystalline specimens, the lth order basal reflection is simply represented by $00l$ reflection. Line profiles were obtained with a CGR X-ray diffractometer (Compagnie Générale de Radio-logie) with Cu radiation. A system of fine slits where the heights of the slits had been reduced was employed to minimize the umbrella effect (Nelson, 1955). Since the effect of the angular separation, $K\alpha_1 - K\alpha_2$, on the line profile is serious and since there is no available means separating them experimentally in the range of angles to be investigated, $CuK\beta$ radiation from unfiltered Cu radiation was used. The relative intensity ratio of $K\alpha_1$ (1·54051 Å): $K\alpha_2$ (1·54433 Å): $K\beta_1$ (1·39217 Å): $K\beta_2$ (1·38102 Å) at the copper target surface is $100:46:15\cdot8:0\cdot15$ (Compton and Allison, 1963). Unlike $K\alpha_1$ and $K\alpha_2$, the contribution of $K\beta_2$ to the $K\beta_1$ is negligible, and the use of $CuK\beta$ would eliminate the intricate correction for the deformation of line profiles due to the doublet $K\alpha_1 - K\alpha_2$.

The theory required two successive $00l$ reflections, and the 002 (5 Å) and 003 (3·33 Å) reflections were chosen for the following reasons: (1) It was almost impossible to eliminate the umbrella

effect from the 001 reflection; (2) the 002 and 003 reflections by $K\beta_1$ radiation were sufficiently separated from the equivalent $K\alpha$ reflections that there are no overlaps at the bases of the peak; and (3) The intensity of the 004 reflection was too weak to be used practically.

The intensity measurements were done by an electronic counting technique at intervals of 0·005° (θ) where the intensities were higher than about a half-peak height, and at intervals of 0·01° (θ) else-where. The counting time was selected so that the statistical error of the counting remained less than 1 per cent. Line profiles for analysis were constructed by plotting the intensity data against diffraction angles. After checking that each line profile was symmetrical, the right-hand side of each line profile was reproduced against the reciprocal function, $t = d^* = 1/d = 2\sin\theta/\lambda$ and the resulting curves were followed by normalization and Fourier transform according to the procedure summarized in the preceding section. These computations were carried out with an electronic computer.

RESULTS AND DISCUSSION

Figure 1 shows the normalized line profiles of the 002 and 003 reflections, which were obtained by equalizing the area of 002 reflection to that of 003 reflection. The normalized line profiles corres-pond to the function $G_{00l_0}(t)$ from which the Fourier coefficients $A_n(00l_0)$ were determined. The coeffici-ents were normalized to the condition $A_0(00l_0) = 1$ and plotted vs. the harmonics n (Fig. 2). Figure 3 shows the difference between the Naperian logar-ithms of A_n (002) and A_n (003), as a function of n. The difference changed almost linearly with n with each specimen except for slight harmonic varia-tions, and the values of $\overline{\delta^2}$ were calculated from the slopes of the straight lines according to equation (9). These values of $\overline{\delta^2}$ were; 0 for specimen N 0·00926 for specimens Y and M and 0·0358 for specimen S.

The probabilities, H_n, were calculated from equation (8) using $\overline{\delta^2}$ and the Fourier coefficients A_n (002), since the approximation used for the derivations was more nearly valid when the lower l_0 index was used. The average number of layers, $\bar{N}$, was evaluated from the slope of the linear portion of the variation curve for H_n, where n was small (Fig. 4). The reciprocals of the slopes for the four specimens gave values between 22 and 28 elementary layers for the average number of layers $\bar{N}$. Although the instrumental broadening could affect the values of H_n, it is probable that all speci-mens consist of crystallites whose average thick-nesses are all about the same.

By definition, the parameter $\overline{\delta^2}$ is the mean square of the variation of the interlayer-spacings.

Table 1. Chemical data of four microcrystalline muscovites with brief features of their X-ray data [numbers of ions on the basis of $O_{10}(OH_2)$]

Specimen		N	Y	M	S
Locality		Nasu, Tochigi Japan	Yoji, Gumma Japan	Miyori, Tochigi Japan	Shiraishi, Tochigi Japan
References		Kodama and Brydon (1968)	Kodama (1957)		Kodama (1965)
SiO_2		46·69	47·65	47·86	48·21
TiO_2		0·07	0·10	0·12	none
Al_2O_3		36·30	37·03	35·90	35·40
Fe_2O_3		} 1·17	0·01	} 0·52	} 1·53
FeO			tr.		
MnO		none	tr.	0·01	n.d.
MgO		0·37	0·04	0·41	0·36
CaO		0·03	tr.	0·09	0·78
K_2O		9·57	9·02	9·42	8·00
Na_2O		0·43	0·76	0·34	0·46
$H_2O(+)$ 110°–1000°C		4·52	4·97	4·60	4·78
$H_2O(-)$ < 110°C		0·26	0·73	0·76	0·96
P_2O_5		n.d.	0·02	n.d.	n.d.
Total		99·41	100·33	100·03	100·48
Tet.	Si	3·087	3·133	3·156	3·164
	Al	0·913	0·867	0·844	0·836
Oct.	Al	1·916	2·002	1·945	1·901
	Fe^{3+}	0·057		0·025	0·075
	Mg	0·036	0·004	0·040	0·035
	$\Sigma_{Oct.}$	2·009	2·006	2·010	2·011
Int.	K	0·846	0·756	0·792	0·669
	Na	0·055	0·097	0·044	0·058
	Ca	0·006		0·006	0·055
	$\Sigma_{Int.}$	0·907	0·853	0·842	0·782
Type of polymorph		$2M_1$	$2M_1$	$2M_1 + 1M$	$2M_1 + 1M$
	$d(00l)(Å)$	$10·00_6$	$10·01_2$	$10·01_2$	$10·04_4$
	$b(Å)$	8·989	8·992	8·993	8·994

The variation may be caused by, (1) lattice distortions within the layer of muscovite, (2) an imperfect relationship between nearly perfect layers of muscovite and (3) a combination of both imperfections. There is no direct way to determine which alternative holds and the relationship of $\overline{\delta^2}$ to the crystallochemical data was explored.

A good linear relation was obtained when the values of $(\overline{\delta^2})^{1/2}$ were plotted vs. the total number of interlayer cations $(\Sigma_{Int.})$ as shown in Fig. 5. This correlation suggests that the observed distortions can be attributed mainly to non-uniform interlayer spaces rather than to lattice imperfections within silicate layers themselves.

SUMMARY AND CONCLUSIONS

(1) The theory developed by Maire and Méring was applied to the analysis of X-ray diffraction line profiles of basal reflections of four microcrystalline muscovites. The method did not require a correction for instrumental broadening which eliminated the major weakness of the previous study.
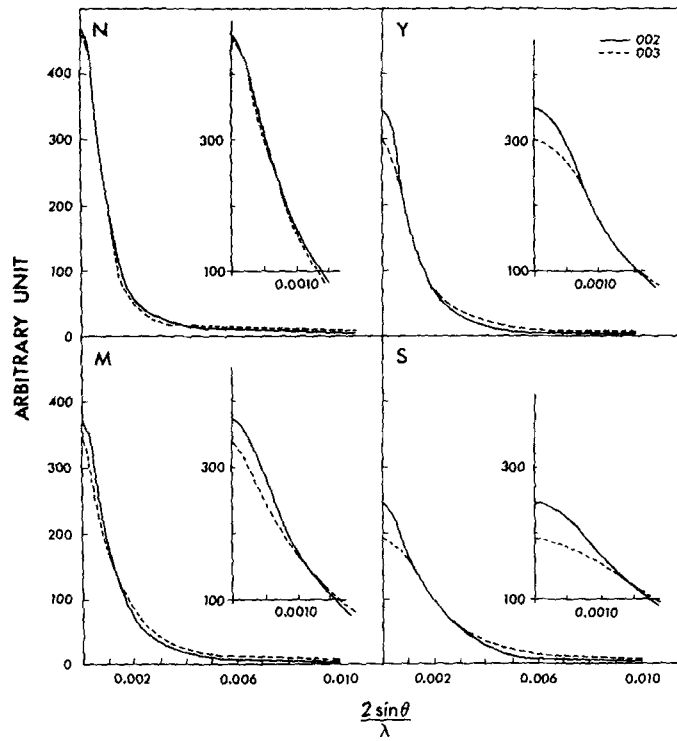
Fig. 1. Normalized line profiles of 002 and 003 reflections of four microcrystalline muscovites.
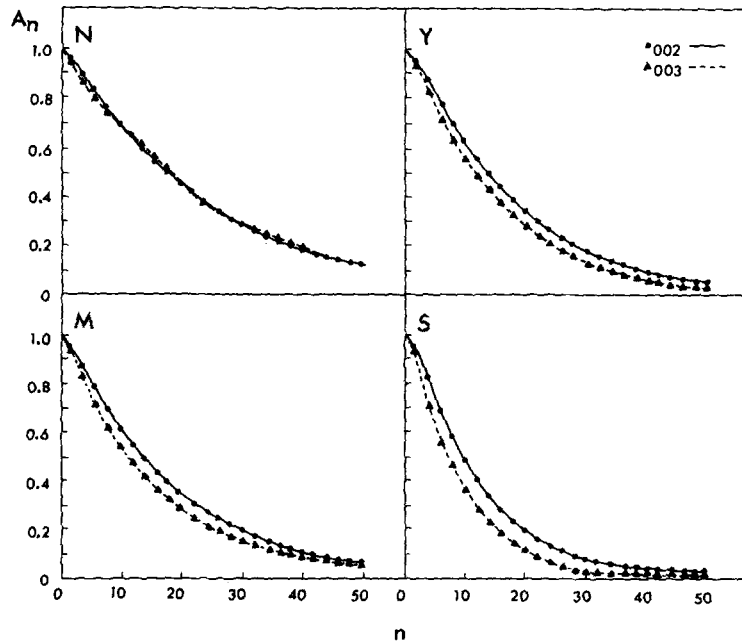
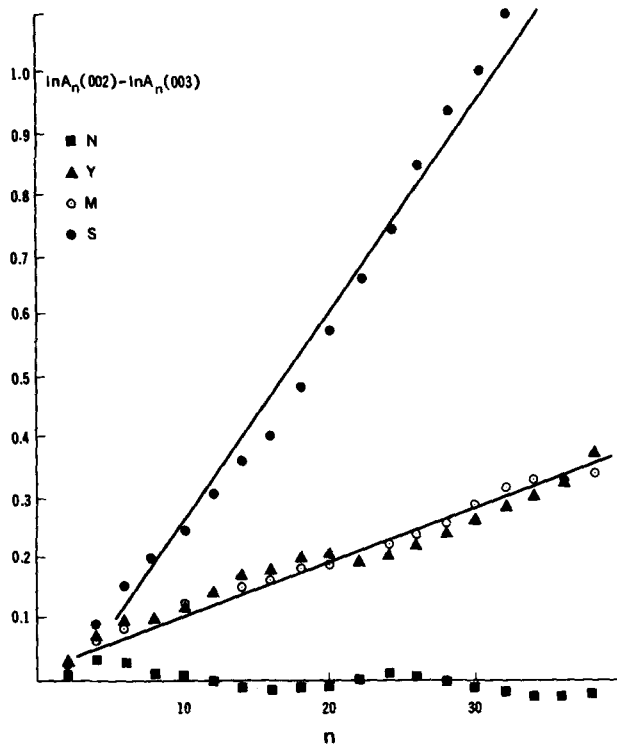Fig. 2. Fourier coefficients A_n for the two basal reflections.

Fig. 3. Difference between Naperian logarithms of A_n (002) and A_n (003).

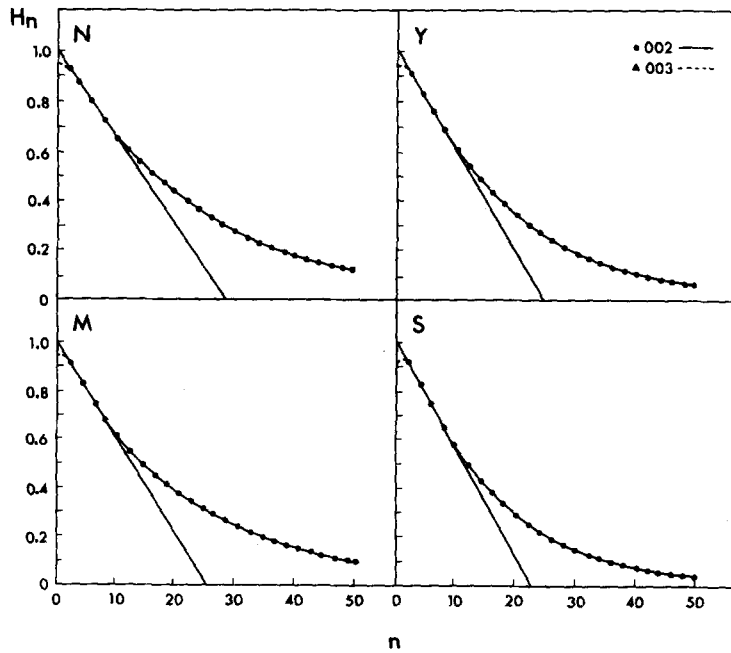
Fig. 4. Plots of probabilities H_n and slopes of the linear portions of the H_n curves for the evaluation of $\bar{N}$.
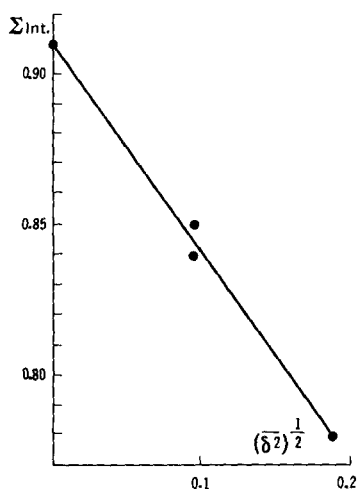
Fig. 5. Interrelation between total number of interlayer cations and square roots of $\overline{\delta^2}$.

(2) The mean square of the variation of interlayer spacings, $\overline{\delta^2}$, i.e. the deviation from the ideal muscovite d_{001} spacing, was 0 for specimen N, 0·00926 for specimens Y and M and 0·0358 for specimen S.

(3) The values, $(\overline{\delta^2})^{1/2}$ increased linearly with a decrease in the total interlayer cations, which suggested that the observed distortions could be attributed mainly to non-uniform interspaces between silicate layers which might have been due to an irregular distribution of interlayer cations.

(4) Since it is reasonable to assume that the effect of instrumental broadening on the line profiles of the basal reflections of the same order was uniform over the specimens, the estimated

values for the mean number of layers $\bar{N}$ could be treated as relative figures. Thus the data indicated that crystallites of all specimens consisted probably of similar numbers of layers.

(5) In order to test the hypothesis deduced from the results (3) above, the line profile analysis of a wider range of samples is required.

(6) In view of the difficulty in obtaining data concerning structural disorder (see Mitra, 1963), this rather simple procedure of the line profile analysis could provide useful and interesting results.

REFERENCES

Compton, A. H. and Allison, S. K. (1963) *X-rays in Theory and Experiment.* 6th Edn., Van Nostrand, Princeton, New Jersey.

Houska, C. R. and Warren, B. E. (1954) X-ray study of the graphitization of carbon black: *J. Appl. Phys.* **35**, 1503–1509.

Kodama, H. (1965) Crystal distortion of sericite: *Clay Sci.* **2**, 121–131.

Kodama, H. (1957) Sericite from Ozawa-mura, Gumma Prefecture: *Mineral J.* **2**, 151–161.

Kodama, H. and Brydon, J. E. (1968) Dehydroxylation of microcrystalline muscovite: *Trans. Faraday Soc.* **64**, 3112–3119.

Maire, J. (1967) *Recherche sur le phénomène de la graphitation.* Thèse pour le Doctrat ès Sciences Physiques, Univ. Paris, Faculté des Sciences N° A-O.1350.

Maire, J. and Méring, J. (1960) *Proc. 4th Conference on Carbon*, p. 345, Pergamon Press, New York.

Méring, J. (1949) L'interférence des rayons X dans les systèmes a stratification désordounée. *Acta Crystallogr.* **2**, 371–377.

Mitra, G. B. (1963) Structure defects in kaolinite: *Z. Kristallogr.* **119**, 161–175.

Nelson, J. B. (1955) In *X-ray diffraction by polycrystalline materials*, p. 96, The Institute of Physics, London.

Résumé – Des études antérieures portant le profil des réflexions basales des muscovites microcristallines ont été raffinées à l'aide d'une adaptation de la méthode développée par Maire et Méring. Afin d'évaluer la variation des espacements interfeuillets, la méthode nécessite seulement la connaissance des valeurs relatives des coefficients de Fourier, sans correction pour l'élargissement instrumental, ce qui était précédemment la source d'un des problèmes les plus critiques. Etant donné que les difficultés associées à la séparation des raies $K\alpha_1$ et $K\alpha_2$ n'ont pas pu être surmontées d'une façon satisfaisante, on a utilisé, pour enregistrer les profils, la raie $K\beta$ au lieu de la raie $K\alpha$.

Les résultats ont confirmé à nouveau que l'élargissement des réflexions 001 n'est pas dû seulement qu'à un effet particules de petite taille, mais aussi à des désordres de la structure impliquant la variation des espacements interfeuillets. Pour les quatre échantillons étudiés, les carrés moyens de la variations des espacements interfeuillets varient de 0 à 0,0358; les racines carrés des espacements sont inversement proportionnelles au nombre total de cations interfeuillets. On considère que les distorsions observées sont essentiellement dues à l'existence, entre les couches de silicate, d'espaces interfeuillets non uniformes provenant d'une distribution irrégulière des cations interlamellaires. Les résultats indiquent également que les cristallites des quatre échantillons sont formés par un nombre similaire de feuillets. La méthode apparaît prometteuse pour l'étude de la nature et de l'étendue des désordres de la structure des micas ou d'autres phyllosilicates.

Kurzreferat – Frühere Untersuchungen der Linienprofile der Basisreflexionen von mikrokristallinen Muskowiten wurden durch Anwendung der Methode nach Maire und Mering weiter verfeinert. Zur Beurteilung der Änderung von Zwischenschichtenabständen erforderte die Methode nur Relativ-

werte von Fourier Koeffizienten, ohne Korrektur für instrumentale Verbreiterung die bisher die Quelle für eines der kritischesten Probleme darstellte. An Stelle der $K\alpha$ Strahlung wurde $K\beta$ Strahlung zur Aufzeichnung der Linienprofile verwendet, da Schwierigkeiten in Verbindung mit der Trennung der $K\alpha_1$ und $K\alpha_2$ Strahlungen nicht auf befriedigende Art bewältigt werden konnten.

Die Messwerte bestätigten, dass die Linienverbreiterung der 00l Reflexionen nicht nur eine Folge der Wirkung kleiner Teilchengrössen war, sondern auch auf strukturelle Störungen im Zusammenhang mit der Änderung der Zwischenschichtabstände zurückzuführen war. Für die hier untersuchten vier Proben bewegten sich die Durchschnittsquadrate der Änderung der Zwischenschichtabstände von 0 bis 0,0358, deren Quadratwurzeln der Gesamtzahl der Zwischenschichtkationen umbekehrt proportional waren. Es wird angenommen, dass die beobachteten Abweichungen hauptsächlich ungleichmässigen Zwischenschichtabständen zwischen Silikatschichten, die sich aus der unregelmässigen Verteilung der Zwischenschichtkationen ergeben, zuzuschreiben sind. Die Messwerte ergaben ferner, dass die Kristallite aller vier Proben aus einer ähnlichen Zahl von Schichten bestanden. Die Methode erscheint vielversprechend zu sein für die Untersuchung des Wesens und des Ausmasses von strukturellen Störungen in Glimmern und anderen Schichtsilikatmineralen.

Резюме — Предыдущие исследования профилей линий базальных отражений от микрокристаллических мусковитов были уточнены с применением метода, разработанного Мэром и Мерингом. Для оценки характера изменений межслоевых расстояний метод требует лишь знания относительных значений коэффициентов Фурье без поправки на инструментальное расширение линии, которая ранее создавала одну из наиболее существенных трудностей. Для регистрации профиля линии вместо излучения $K\alpha$ использовалось излучение $K\beta$, так как затруднения, связанные с разрешением излучения $K\alpha_1$ и $K\alpha_2$, не могут быть полностью преодолены.

Полученные данные вновь подтвердили, что расширение рефлексов (002) обусловлено не только влиянием малого размера частиц, но также и структурным разупорядочением, включающим непостоянство межслоевых расстояний. Для четырех изученных образцов средний квадрат отклонения межслоевых расстояний изменялся от 0 до 0,0358, причем квадратный корень из этой величины обратно пропорционален общему числу межслоевых катионов. Сделан вывод о том, что наблюдаемые искажения связаны, главным образом, с неоднородностью расстояний между силикатными слоями, обусловленной неупорядоченным распределением межслоевых катионов. Полученные данные указывают также на то, что кристаллиты во всех четырех изученных образцах состоят из примерно одинакового количества слоев. Метод обещает оправдать себя при изучении природы и степени структурного беспорядка в слюдах и других слоистых силикатах.