

STRUCTURAL FACTORS CONTROLLING STACKING SEQUENCES IN DIOCTAHEDRAL MICAS

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Abstract—Deviations in the symmetry of mica single layers from the ideal arrangement with all atoms in hexagonal arrays are caused by the structural factors such as compositional variation (substitutions), order-disorder, and distortions. The derivative crystal structure theory as applied to mica polymorphism, implies that the ideal symmetry of the mica single layer ($C2/m$) can be altered to one of its standard subgroups by these structural factors. The “derivative structure” of the single layer may allow a unique interlayer and a specific intralayer configuration and result in a regular stacking sequence.

INTRODUCTION

DIFFERENT stacking sequences of topologically similar mica single layers may result in superstructures known as mica polymorphs for a given composition. Studies on synthetic and natural micas from different environments by Hendricks and Jefferson (1939), Heinrich and Levinson (1955), Smith and Yoder (1956) indicated that structural forces mainly due to compositional variation of micas control the stacking sequence. Smith and Yoder (1956) presented a possible mechanism of different mica stacking sequences as a growth phenomenon in the following way: Micas may grow by “screw dislocation mechanism” as a real crystal or by “secondary nucleation” like a perfect crystal. In the latter case structural control has to be effective for formation of a regular stacking sequence. In the former case, however, no structural control is necessary because the spiral growth has its memory. The origin of dislocations in the spiral growth mechanism was, however, not questioned. Bradley (1957), and Steinfink (1962) suggested that (Si, Al) ordering may create structural forces controlling stacking sequences.

Radoslovich (1960, 1963) has extensively analyzed the crystal-chemical aspects of layer silicates in the light of Pauling’s rules for ionic structures. He emphasized the role of the interlayer cation in the b -dimension of micas and the ditrigonal surface configuration of basal oxygens. He suggested that “local balance of forces” in layer structures may provide a means of structural control over polymorphism. Takéuchi (1966) suggested that the structural control may be related to the direction of O–H bonds in mica single layers.

Zyvgin (1962, 1967) developed a theory of mica polymorphism based on close packing of anions.

Polymorphism and polytypism

If the structural differences between two mica modifications (e.g. $2M_1$ and $3T$) consist only of a different stacking sequence, the single layers in both structures being equivalent, then this may be considered as a one-dimensional polymorphism, as observed in the structures of SiC and ZnS. This kind of polymorphism is called “polytypism”, a recent account of which is presented by Verma and Krishna (1966). Accordingly, a large number of factors interplay in the phenomenon of polytypism: temperature, crystallization rate, impurities, growth dislocations and thermal vibrations of the structure. Although theories advanced so far are unable to explain satisfactorily all the observed polytypes, factors controlling them do not seem to be strong, because several stacking sequences are commonly present within a single sample. If the single mica layers were ideal or close to it, i.e. all atoms in hexagonal arrays with completely disordered tetrahedral and octahedral cation distributions, we could expect a large variety of stacking sequences which would correctly be called mica polytypes. Generally, single mica layers display deviations from the ideal arrangement, which may create additional structural factors affecting the stacking sequence. The changes in mica single layers with the ideal symmetry $C2/m$ may be caused by compositional variation (substitutions), order-disorder, and distortions. Upper and lower tetrahedral layers can be linked in six different ways in an ideal single mica layer; this, combined with six different superpositions of two such single layers, gives rise to polymorphism in these minerals. If a certain intralayer configuration (i.e. intralayer shift due to linkage of two tetrahedral sheets) and a specific interlayer configuration become

energetically favorable for a mica, we may then expect a regular stacking sequence. The characteristics of single layers and the resulting interlayer configurations in the natural micas with regular stacking sequences may then reveal the structural factors controlling polymorphism in micas. In the following, structural details obtained from the recent refinements of $2M_1$ Georgia muscovite and $2M_1$ phengite (Güven, 1971) and $3T$ muscovite (Güven and Burnham, 1967) will be examined for this purpose.

Intralayer configuration

As seen from Figs. 1b and 1c, in $2M_1$ Georgia muscovite and $3T$ muscovite with Al^{3+} as the major octahedral cation, apical oxygens and OH-ions are significantly displaced from their ideal positions shown in Fig. 1a. The O_a-O_b-OH configuration around an occupied site becomes quite distinct from the other such triads in octahedral sheets. The linkage of upper and lower tetrahedral

sheets is centrosymmetrical in $2M_1$ muscovite (Fig. 1d). If, however, tetrahedral cations are ordered over the two tetrahedral sites, the above arrangement will then be less stable, for octahedral polyhedra will have two oxygens with unbalanced electrostatic charges along a shared edge (O_a-O_a' or O_b-O_b'), which is extremely shortened in dioctahedral micas because of octahedral vacancies. Therefore, the arrangement found in the $3T$ muscovite structure with a two-fold axis relating upper and lower tetrahedral sheets seems to be more stable (Fig. 1e) for it avoids the occurrence of two apical oxygens with unbalanced electrostatic charges along the shared edges.

The basal and apical oxygen hexagons are similarly distorted into pseudo ditrigonal rings in both $2M_1$ and $3T$ muscovite structures. Basal oxygens are not coplanar; one of them is displaced off the plane of the other two basal oxygens by an amount Δz that we consider as the amount of tetrahedral tilt. It is important to remember that tetrahedral

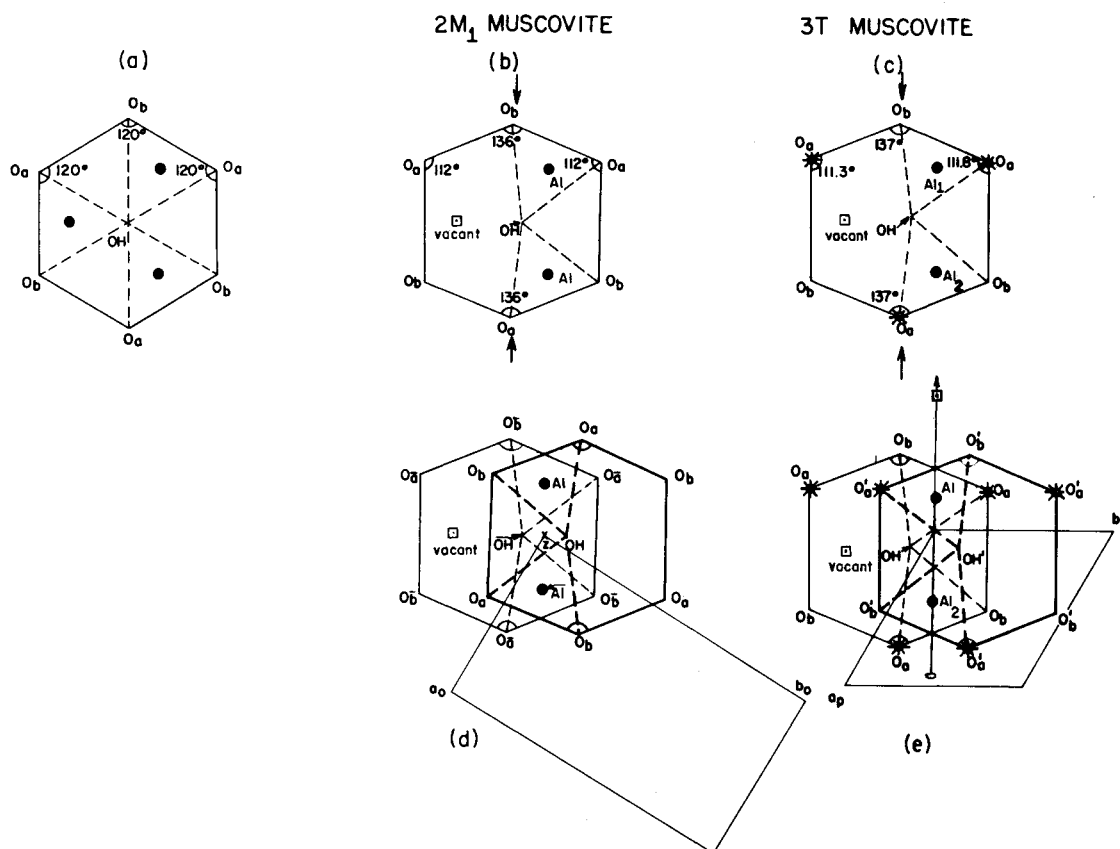


Fig. 1. Octahedral configuration in $2M_1$ and $3T$ muscovite structures projected on (001). Circle with spokes represents apical oxygen of Al-containing tetrahedra in case of (Si, Al) ordering.

tilt is somewhat different for $2M_1$ muscovite ($\Delta z = 0.22 \text{ \AA}$) than for $3T$ muscovite ($\Delta z = 0.14 \text{ \AA}$). As explained in detail by Takéuchi (1966), tetrahedral tilt causes corrugations or ripples in basal oxygen planes. These corrugations strike parallel to the direction of intralayer shift, namely $[\bar{1}10]$ direction in the first mica layer of the $2M_1$ cell (Fig. 2) and parallel to the $[01.0]$ direction in the first mica layer of the $3T$ cell. Thus a key structural factor in controlling the stacking sequence seems to be that the intralayer shift is parallel to the strike of tetrahedral corrugations in dioctahedral micas.

Interlayer configuration

The relative orientation of two adjacent mica layers with respect to each other can be described by one of the six twofold axes shown in Fig. 2. The structural changes caused by substitutions and distortions in real mica single layers may, however, make some of these six twofold axes energetically more favorable, as will be explained in the following discussion.

The electrostatic charge imbalance of the basal oxygens will vary from zero to $n/4$, where n is the number of trivalent cation (Al^{3+} , Fe^{3+}) proxying for Si^{4+} per formula unit in micas. In the ideal hexagonal arrangement of basal oxygens, the interlayer cation would be in a hexagonal prism co-

ordination with six oxygens above and six oxygens below it, with an expected K-O distance of 3.04 \AA . The effective ionic radius of such a cation is 1.68 \AA , which is exceedingly larger than the effective ionic radius of K and other interlayer cations in micas. Moreover, the charge of a monovalent interlayer cation (K^+ , Na^+) is not sufficient to satisfy all the unbalanced charges of the twelve basal oxygens. From the "packing" consideration this represents an open packed arrangement of basal oxygens in the interlayer region. In the analyzed structures of dioctahedral micas, $2M_1$, $3T$ muscovite, $2M_1$ margarite and $2M_1$ phengite, the interlayer cation is closer to six basal oxygens (three above and three below) forming a coordination polyhedron close to an octahedron (Fig. 2). The relationships between the interlayer separation (η), the inward movement of basal oxygens (Δ_{in}) in the (001) plane towards the interlayer cation, the tetrahedral rotation (α), the length of interlayer cation oxygen bonds (d_i) and the size of tetrahedral edge (l_t) can be expressed in the following way:

$$d_i^2 = \left[\frac{\eta}{2} \right]^2 + (l_t - \Delta_{\text{in}})^2 \quad (1)$$

$$\Delta_{\text{in}} = [(\sqrt{3}/3) \sin \alpha + 1 - \cos \alpha] \cdot l_t \text{ (Donnay } et al. \text{ 1964a) } \quad (2)$$

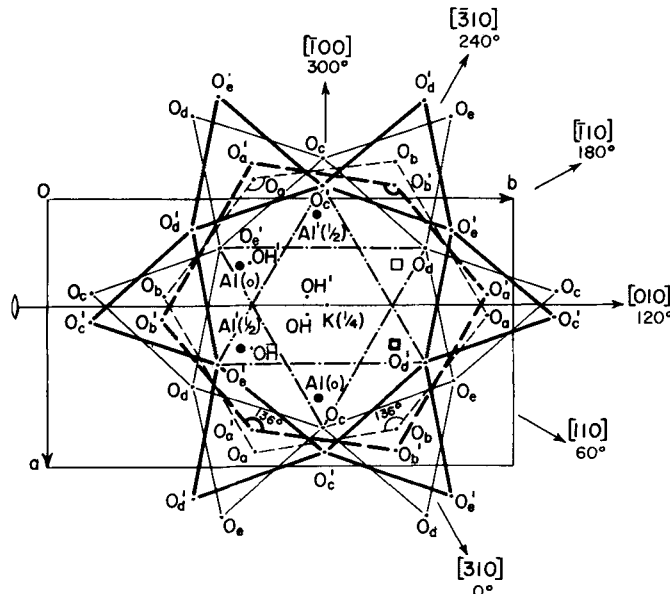


Fig. 2. Interlayer configuration in $2M_1$ muscovite projected on (001). Thick solid and dashed lines: tetrahedral sheets above the interlayer cation (K). Thin solid and dashed lines: tetrahedral sheets below K. Dashed-dot lines: coordination polyhedron of interlayer cation. Letters with a prime, e.g. O_a' , indicate that this atom is related by the twofold axis parallel to $[010]$ to the atom O_a .

Note. Rotation angles indicated on the figure are only valid if the single mica layer possesses $C2/m$ symmetry.

or as an approximation: $\Delta_{in} \cong (1/\sqrt{3}) \cdot l_t \cdot \tan \alpha$ (3)

Interlayer separation is related to ψ and d_i by the expression:

$$\eta = 2d_i \cdot \cos \psi \quad (4)$$

where ψ is the angle between c^* and a K–O direction (flattening angle of the octahedron around the interlayer cation).

From the observed interlayer separation, 3.24 Å for $2M_1$ muscovite and 3.25 Å for $2M_1$ phengite, ψ 's are calculated as 55°26' for $2M_1$ muscovite and 55°18' for $2M_1$ phengite, as opposed to its ideal value of 54°44' for a regular octahedron. Thus, the interlayer cation has a trigonal antiprism coordination close to a regular octahedron with the basal oxygens. Hence the basal oxygens from top and bottom are rather in "closed packed" than in "open packed" arrangement in the interlayer region. The basal oxygen-interlayer cation attractions are in general weak for monovalent interlayer cations and small amounts of $Al^{3+} \rightarrow Si^{4+}$ substitutions. From Equation (1) we may infer that d_i is largely dependent on Δ_{in} ; hence, on tetrahedral rotations. The latter are largely caused by the dimensional misfit between octahedral and tetrahedral sheets, therefore, mainly by the substitutions in the octahedral and tetrahedral sites in mica single layers. These rotations will be opposed by interlayer cations only if they start causing a large interlayer separation (η). In $2M_1$ and $3T$ muscovite the surface network is pseudotrigonal and K is displaced from the center of the basal oxygen configuration along [010] direction. In both micas the K–O distances are very similar and the deviations (2α) of interatomic angles between basal oxygens from the ideal 120° are similar, i.e. similar tetrahedral rotations. Thus, the surface configuration of the oxygens is similar in $2M_1$ and $3T$ muscovite.

Top and bottom basal oxygens (hence, the tetrahedral sheets) around the interlayer cation can be so arranged as to cause least strains in the interlayer region. The basal oxygens are highly polarized by small tetrahedral cations with large charges like Si^{4+} and Al^{3+} . Superposition of these oxygens in an ideal open packed arrangement causes a large amount of repulsion in the interlayer region. Since the two tetrahedral sheets exert forces upon the interlayer cation in opposite directions, a stable equilibrium might be possible if the top and bottom tetrahedral sheets were arranged symmetrically with respect to interlayer cations, e.g. by a center of symmetry or by one of six twofold axes or more symmetrical, e.g. $2/m$. An interlayer configuration with an inversion center at the interlayer cation seems to be most stable both from packing

and electrostatic force considerations. There is, however, one significant factor which makes this arrangement less stable for dioctahedral micas; namely, the corrugations of the bottom tetrahedral sheet, parallel to $[\bar{1}10]$ in $2M_1$ cell, will not mesh, but rather clash with the corrugations of the top tetrahedral sheet. This causes a larger amount of oxygen–oxygen repulsion in the interlayer region. This kind of repulsion has to be interpreted as electrostatic repulsive forces, which will also be effective to some extent even when the opposing oxygen surfaces are not in direct contact as in dioctahedral micas. These corrugations caused by tetrahedral tilts are characteristic features of dioctahedral mica single layers, though more pronounced in $2M_1$ muscovite ($\Delta z = 0.22$ Å) than in $2M_1$ phengite ($\Delta z = 0.16$ Å) and $3T$ muscovite ($\Delta z = 0.14$ Å). Thus, because of tetrahedral tilt the centrosymmetrical interlayer configuration becomes less favorable in dioctahedral micas.

Three of the six possible twofold axes, parallel to [110], $[\bar{1}10]$, and $[\bar{1}00]$ directions* in the $2M_1$ cell (Fig. 2) are not favorable, for they almost form a trigonal prism as a first coordination around K and create a closer superposition of anions and cations of the two tetrahedral layers in projection parallel to c^* (Fig. 2). The other three twofold axes, parallel to [310], [010], and $[\bar{3}10]$ directions† seems to be equally favorable at first inspection of Fig. 2. In fact, the twofold operation parallel to [310], (corresponding to 0° rotation for mica single layers with $C2/m$ symmetry), would create an arrangement in which octahedral vacancies are related centrosymmetrically with respect to the interlayer cation. As explained in the case of the interlayer configuration with an inversion center, the clash of corrugations in top and bottom basal oxygen planes will make this twofold axis less favorable. The twofold axes parallel to [010] and $[\bar{3}10]$ however, relate the tetrahedral corrugations in the bottom and top layers around K by 120° and 240° to each other, allowing least oxygen–oxygen repulsion. These two operations (corresponding to 120° and 240° rotations for mica single layers with $C2/m$ symmetry) are therefore most probable for dioctahedral micas. Inspection of Fig. 2 suggests there is no obvious structural reason to prefer the [010] direction over the $[\bar{3}10]$ direction. As long as one of them is consistently chosen, the resulting stacking sequence will be the same and the structures will

*These operations correspond to 60°, 180°, and 300° rotations of layers if the mica single layer possesses $C2/m$ symmetry.

†These operations correspond to 0°, 120°, and 240° rotations of layers if the mica single layer possesses $C2/m$ symmetry.

be enantiomorphs of each other. The choice of the [010] direction as the twofold symmetry axis was explained by Radoslovich (1960, 1963) by the interactions between K and O_a apical oxygens on the premises that the latter have higher unsatisfied charges because of (Si, Al) ordering in tetrahedra of $2M_1$ muscovite. Since then, however, it has been shown that such an ordering does not exist (Gatinéau, 1963; Burnham and Radoslovich, 1964; Güven, 1971).

The interlayer configuration with a twofold axis parallel to [010] in the $2M_1$ cell combined with a single layer with $C\bar{1}$ symmetry will establish periodicity after two 10 Å layers as in $2M_1$ muscovite. The interlayer configuration with the same symmetry, as in $2M_1$ muscovite, combined with a single layer with $C2$ symmetry will, however, attain periodicity after three 10 Å layers as in $3T$ muscovite. These two muscovite modifications, having very close chemical compositions, indicate that the difference in their structures is not only the stacking sequence but also in the symmetry of their single layers; hence, they are from the structural viewpoint polymorphs but not polytypes (Güven and Burnham, 1967).

The crystal structure of $2M_1$ phengite showed that there is a tendency for tetrahedral cation ordering, and if the order further increases, e.g. at lower temperatures, this may create a different stacking sequence, possibly $3T$. With increasing Mg content in octahedra, the dimensional "misfit" between tetrahedral and octahedral sheets is decreased; hence, the tetrahedral sheets are less corrugated and therefore less effective in controlling the intralayer shift. In addition, the increased silica content in tetrahedra will decrease the interlayer cation-basal oxygen interactions, and relative positions of top and bottom tetrahedral sheets will then be decided by the forces due to second neighbors (apical oxygens and octahedral ions) of the interlayer cation. These forces will, therefore, not be as strong in tetrasilicic micas as in muscovite.

Trioctahedral micas

At this point it is worth mentioning certain characteristics of single layers of trioctahedral micas, whose stable form seems to be the $1M$ stacking sequence. Refinements of crystal structures of $1M$ trioctahedral micas, synthetic ferriannite (Donnay *et al.*, 1964b), ferriphlogopite (Steinfink, 1962), xanthophyllite (Takéuchi, 1966) and lithian fluorphlogopite (Takéda *et al.*, 1966), show that the single layer possesses $C2/m$ symmetry, which is the symmetry of the ideal mica layer. Distortions, which are larger in xanthophyllite than in any dioctahedral mica, and octahedral ordering in trioctahedral micas preserve that symmetry. Basal

oxygens form ditrigonal rather than hexagonal arrays having $2/m$ symmetry; hence the stable centrosymmetrical arrangement around the interlayer cation. The twofold axis parallel to [010] in $1M$ cell, passing through the interlayer cation, corresponding to a 0° rotation, is included in this symmetry.

Although the other two possible twofold axes, parallel to the directions $[310]$ and $[3\bar{1}0]$ in $1M$ cell, corresponding to 120° and -120° rotations, also conserve the inversion symmetry between tetrahedral sheets around the interlayer cation, the octahedral cations above the interlayer cation are no longer centrosymmetrically related to those below the interlayer cation, unless the three octahedral cations are equivalent. Due to second or higher neighbor interactions, the 0° rotation in the interlayer configuration seems slightly more favorable than the $\pm 120^\circ$ rotations. It is obvious that the two other rotations can easily occur and cause irregular stacking sequences.

Derivative crystal structure theory and mica polymorphism

Derivative crystal structure theory deals with the symmetry relation between a basic structure and the structure derived from it by suppression of certain symmetry elements (including translation) of the original structure (Buerger, 1947). Because the suppression of symmetry elements of the original crystal structure is caused by substitutions (compositional variation), order-disorder and distortions, Buerger's derivative crystal structure theory is useful for understanding crystallographic aspects of mica polymorphism.

The ideal symmetry of a mica single layer is $C2/m$ and the above factors may alter this symmetry to one of the derivative symmetries of this "basic" symmetry. Referred to the same cell of a single layer, these derivative symmetries are: $C2$, Cm , $C\bar{1}$, $C1$, $P2$, Pm , $P\bar{1}$, and $P1$. The ideal interlayer configuration in micas possesses $6/m\ 2/m\ 2/m$ symmetry which creates the largest amount of oxygen-oxygen repulsion in the interlayer, since it superposes these highly polarized oxygens exactly on top of each other. From the "packing" consideration this is the most open packed interlayer arrangement. In most of the natural micas the interlayer configuration is so distorted due to structural changes in mica single layers that oxygens, highly polarized by small tetrahedral cations, do not superpose and the packing alters more toward a close packed arrangement. Therefore, $6/m$ will be destroyed. From the remaining $2/m$ symmetry of the interlayer configuration, m will only be permissible if it coincides with the m of the two single layers above and below.

There are six possible directions for the twofold axes in the interlayer configuration: referred to the $2M_1$ cell those parallel to $[110]$, $[\bar{1}10]$, and $[\bar{1}00]$ do not alter the oxygen–oxygen superpositions as much as the other three twofold axes parallel to the $[310]$, $[010]$, and $[\bar{3}10]$ directions (Fig. 2). Thus, the last three twofold axes do occur in most of the natural micas. An interlayer configuration with an inversion center at the interlayer cation is both from packing and electrostatic force considerations the most stable arrangement. This inversion operation will include the $[310]$ twofold axis if the single layer possesses $C2/m$ symmetry. If, however, considerable tetrahedral tilt is present, the interlayer configuration with a twofold axis parallel to $[010]$ or $[\bar{3}10]$ will be more favorable as observed in dioctahedral micas. The different mica polymorphs may then be generated by the combination of the symmetry of the interlayer configuration with the derivative symmetry of the single layer as shown in the following examples:

Single layer	Interlayer	
$(C2/m)$	$(\bar{1})$	$= 1M$
$(C2)$	$(2[010])$	$= 3T$
$(C\bar{1})$		
$(C\bar{1})$	$(2[010])$	$= 2M_1$

The nature of the single layer, its derivative structure, may make a certain interlayer and a specific intralayer configuration more favorable and result in a regular stacking sequence.

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- Résumé**—Les écarts observés entre la symétrie de feuillets isolés de mica et l'arrangement idéal dans lequel tous les atomes sont rangés selon des hexagones sont dus à des facteurs structuraux tels que la variabilité de la composition (substitution), les effets d'ordre–désordre, et les distorsions. La théorie de la "structure dérivée" du cristal, appliquée au polymorphisme des micas, implique que la symétrie idéale du feuillet isolé de mica ($C 2/m$) peut être altérée en l'un de ses sous groupes normaux par ces facteurs structuraux. La "structure dérivée" du feuillet isolé peut autoriser une configuration inter-feuillet unique et une configuration intrafeuillet spécifique, et entraîner de ce fait une séquence de superposition régulière.
- Kurzreferat**—Abweichungen in der Symmetrie von Glimmereinzelschichten von der idealen Anordnung mit allen Atomen in hexagonalen Verteilungen werden durch strukturelle Faktoren, wie etwa Veränderungen in der Zusammensetzung (Substituierung), Ordnung–Unordnung und Verzerrungen verursacht. Die Theorie der abgeleiteten Kristallstruktur, angewendet auf Glimmerpolymorphie, besagt, dass die ideale Symmetrie der Glimmereinzelschicht ($C2/m$) durch diese strukturellen Faktoren zu einer ihrer normalen Untergruppen verändert werden kann. Die "abgeleitete Struktur" der Einzelschicht kann eine eindeutige Zwischenschicht- oder eine spezifische Intraschichtkonfiguration ermöglichen und eine regelmässige Stapelfolge ergeben.

Резюме — Отклонения симметрии слюдяного слоя от идеального строения с гексагональным мотивом расположения всех атомов, обусловлены рядом структурных факторов, таких как изменения в составе (замещения), порядок-беспорядок, искажения. Теория производных кристаллических структур в применении к полиморфизму слюд показывает, что идеальная симметрия слюдяного слоя ($C2/m$) может изменяться под действием перечисленных структурных факторов до одной из присущих ей стандартных подгрупп. «Производная структура» слюдяного слоя может обуславливать специфическую конфигурацию межслоевого пространства и, как следствие, приводить к упорядоченной последовательности слоев.