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FIRST ANNUAL REPORT

NATURAL MICA STUDIES

(Covering period September 1, 1951, to August 31, 1952)

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INTRODUCTION

During the fourth quarter emphasis in the natural mica studies has been shifted to the phlogopite-boitite series. Due to the success in correlating chemistry with polymorphism in the muscovite-lepidolite series, a similar correlation is being attempted in the phlogopite-biotite series. The structures of over 200 specimens, of these micas, chiefly of pegmatitic origin, have been determined. About 55 of these have been chemically analyzed. The results to date indicate no obvious relationship between chemistry and polymorphism. Further work on this problem appears justified, however, with particular attention placed on the role of temperature and paragenesis in the crystallization history of these micas. During the next quarter, the crystal structure of types from extrusive rocks will be studied, in the hope that some indication as to the effect of temperature upon crystallization will be forthcoming. Work also will be initiated on the paragenesis and optical properties of micas of this series.

Our work on these problems has been greatly facilitated by the cooperation of many scientists throughout the world who have generously supplied analyzed specimens. Particular thanks are due Professor J. Jakob, of the Technical University of Zurich, who has supplied 35 analyzed specimens.

TECHNIQUE

The x-ray study of phlogopites and biotites is more difficult and time-consuming than that of muscovites and lepidolites. A crystal orientation

procedure based on optical methods was perfected and used satisfactorily with the latter series. The positions of the crystallographic axes were determined by means of interference figures and extinction positions, for in muscovite and lepidolite the crystallographic axes correspond essentially with extinction positions. In phlogopite and biotite, however, the birefringence of (001) sections is often nil and 2V approaches zero; thus an optical orientation method is impractical.

To overcome these problems, Laue photographs were used to determine the positions of the desired axes. However, Laue photographs of some monoclinic specimens appear so nearly rhombohedral that the true plane of symmetry is not easily determined. Weissenberg photographs about the two pseudo a-axes were often taken before the one about the true a-axis was obtained. An alternative method was used occasionally, whereby it was not necessary to take a Laue photograph. This consisted of producing a precussion figure on the cleavage flakes and choosing one of three rays at random as the rotation axis, with the hope that it would be the true a-axis and not one of the pseudo a-axes. But, as in the first method, several pictures usually were required before the structure could be determined accurately.

0-level Weissenberg photographs taken about one of the pseudo a-axes of the 2-layer monoclinic polymorph appear identical with the 0-level a-axis photograph of the 6-layer triclinic biotite illustrated (on p. 745) by Hendricks and Jefferson (1939). Likewise, 0-level pseudo a-axis photographs of the 1-layer monoclinic polymorph strongly resemble 0-level b-axis photographs of the 6-layer monoclimic polymorph reported only in lepidolites. Therefore extreme caution had to be practiced in deciding the structure of these micas. Diffuse scattering, particularly among the biotites, also made structure determinations difficult in several instances.

In some specimens the crystal development of the flakes was poor, or the only analyzed specimens available were powders. Under these conditions single-crystal methods were not possible, and the powder x-ray method was employed. It was found that iron radiation was most suitable and even essential in studying biotites by the powder method. A filter was not used, for it increased the necessary exposure time by a factor of nearly three. Unfortunately the powder patterns of the common polymorphic forms, especially the 1-layer monoclinic and 3-layer rhombohedral varieties, are so similar that in several cases no definite structure could be assigned.

THE STRUCTURE OF PHLOGOPITE

The structures of approximately 80 phlogopite crystals from about 50 different specimens (28 chemically analyzed) were determined by means of

the Weissenberg method. The results are impressive in that all but 5 have
crystallized with the 1-layer monoclimic structure. Of those 5, 3 have
crystallized as the 2-layer monoclimic polymorph, whereas the remaining 2
have crystallized with 3-layer rhombohedral structure (Table 1).

TABLE 1. 2- AND 3-LAYER PHLOGOPITES

Number	Location	Reference	Structure
1062 (a and b)	Madagascar	Jakob and Parga-Pondal (1932) No. 65	2-layer monoclinic
1064 (a and b)	Madagascar	Jakob and Parga-Pondal (1932) No. 63	2-layer monoclinic
1261 (b and c)	Madagascar	Mauguin (1928)	3-layer rhombohedral
1220 (a)	Hull, Quebec	-----	2-layer monoclinic
1227 (a)	Labelle Co., Quebec	-----	3-layer rhombohedral

It is noteworthy that three of the above specimens are from Mada-
gascar, but that at least 4 other specimens of Madagascar phlogopite have
crystallized with the common 1-layer monoclinic structure. A third crystal
obtained from the material supplied by Professor Mauguin, No. 1261(a), has
crystallized as the 1-layer monoclinic form; there probably is little dif-
ference in the composition of these several crystals from the same specimen.
The specimens of known composition that have crystallized as the 2- and
3-layer forms do not appear to be chemically aberrant. In fact, the com-
positions of all the Madagascar varieties are practically identical.

A few of the phlogopite patterns show diffuse scattering of varying
intensity. No correlation of diffuse scattering with polymorphism or chem-
istry was obtained.

These findings are in qualitative agreement with those of Hendricks
and Jefferson (1939), who found 14 phlogopites with the 1-layer monoclinic
structure and 1 each with the 2- and 3-layer forms. No new polymorphs of
phlogopite have been found in our investigations. Crystals reported by
Pieruccini (1950) to be triclinic on the basis of goniometric measurements
were isolated from a specimen supplied by him. Weissenberg photographs
indicate the structure is the common 1-layer monoclinic form.

THE STRUCTURE OF MANGANOPHYLLITE

The eight analyzed Swedish manganophyllites described by Jakob (1925) were available for study, as well as a manganophyllite from Idaho described by Kauffman, Mortimore, and Hess (1950). All have crystallized as the 1-layer monoclinic polymorph. The chemical composition of these micas varies appreciably; for instance, total iron oxides range from 0.91 to 16.94%, total manganese oxides from traces to 12.21%, MgO from 21.18 to 29.28%, and Al_2O_3 from 0.00 to 13.84%. The optical properties of these micas also vary; for example, 2V is reported by Jakob (1925) as varying from 3°35' to 33°30', but again it does not appear possible to correlate this variation with changes in chemistry. Several manganophyllites have their optic planes normal to the side pinacoid in contradistinction to the general relationship that micas crystallizing as 1-layer monoclinic polymorphs usually have their optic planes parallel with the side pinacoid.

0-level a-axis Weissenberg photographs of specimens No. 1083 and 1079 are extremely interesting in that at first glance they appear similar to 0-level a-axis photographs of the 3-layer rhombohedral polymorph. However, some reflections are of stronger intensity than would normally be expected. Further study shows that the more intense reflections were obtained by rotation about the true a-axis, whereas the others were obtained from the pseudo a-axes. The total effect of combining these patterns is that of an 0-level a-axis 3-layer rhombohedral polymorph. Powder photographs of all the manganophyllites were taken and proved to be identical, thus further establishing the 1-layer monoclinic structure for all specimens. It is noteworthy, that specimens No. 1083 and 1079, through whose twinning the 3-layer rhombohedral structure is approximated have the smallest 2V (3°35' and 3°40', respectively) of the manganophyllite suite. This suggests a possible relationship between the variable 2V and twinning, rather than between optics and chemistry.

STRUCTURE OF BIOTITES

Although the structures of about 200 biotites have been determined, only a few definite conclusions are available at present. Attempts to correlate chemistry with composition have so far been unsuccessful, but information is available on the geographic and geologic distribution of various polymorphs.

About 80 biotite specimens from about 15 pegmatite deposits in the southeastern United States were studied. A compilation of the results

showed that about 50 have crystallized as the 2-layer monoclinic polymorph, 15 as the 3-layer rhombohedral polymorph, but only 1 as the 1-layer monoclinic polymorph. Heavy diffuse scattering prevented accurate structural determination of the remainder. Diffuse scattering is predominant in biotites from this area. The results of studies from other widely scattered pegmatite districts are summarized below.

TABLE 2. GEOGRAPHIC DISTRIBUTION OF BIOTITE POLYMORPHS

New England - exclusively 1-layer monoclinic forms.

Southern Norway - Predominantly 1-layer monoclinic forms, but 2-layer forms abundant.

Canada, Bancroft, and Wilberforce Districts - predominantly 1-layer forms.

Colorado, Guffey District - predominantly 1-layer forms.

Geological environment may play an important role in the crystallization of biotite micas. The pegmatites of the southeastern United States (North Carolina, particularly) are of quartz dioritic composition, whereas those of the Bancroft area are nepheline syenitic. It seems possible that biotites crystallizing from such different magmas might have structures characteristic of their particular environments, but this hypothesis needs considerable further study. A knowledge of the temperature of crystallization of these micas would be very useful. For this reason a suite of biotites from extrusive rocks believed to have formed under relatively high temperatures will be studied during the next quarter.

In the study of the biotites, the structures of several uncommon varieties were determined. The results are tabulated on the next page:

During this study neither the 6-layer nor the 24-layer triclinic biotites described by Hendricks and Jefferson (1939) were found. Only the 1- and 2-layer monoclinic and the 3-layer rhombohedral polymorphs were definitely established. In a few scattered instances patterns not assignable to any known polymorphs were recorded on Weissenberg photographs. Detailed study of these specimens will probably show that they consist of crystals with mixed structures rather than new polymorphs.

TABLE 3. STRUCTURES OF BIOTITE VARIETIES

732(a)	waddoite	Isle of Waddo	1-layer monoclinic
746(a)	lepidomelane (pterolite)	Brevig, Norway	2-layer monoclinic
752(a)	meroxene	Mt. Vesuvius	1-layer monoclinic
753(a)	meroxene (white)	Mt. Vesuvius	3-layer rhombohedral
759(a)	meroxene	Mendham, N. J.	1-layer monoclinic
754(a)	calciobiotite	Italy	1-layer monoclinic
770(a)	annite	Rockport, Mass.	1-layer monoclinic
1116(a)	annite	Rockport, Mass.	1-layer monoclinic
775(a)	siderophyllite	Pikes Peak, Colo.	1-layer monoclinic
1117(a)	siderophyllite	Brooks Mt., Alaska	1-layer monoclinic (very diffuse scattering)
783(a)	cryophyllite	Rockport, Mass.	1-layer monoclinic
1112(a)	monrepite	Finland	1-layer monoclinic
1113(a)	eukamptite	Presburg, Hungary	1-layer monoclinic
1114(a)	bastonite	Bastogne, Belgium	2-layer monoclinic
1130(a)	haughtonite	Scotland	(?) - mixed structures

STRUCTURE OF HIGH-SILICA SERICITES

Schaller (1950), in interpreting the chemical composition of the high-silica sericites, reached the conclusion that they are intermediate between muscovite and leucophyllite and generally contain an appreciable quantity of a divalent element, usually magnesium. Other micas in the series include phengite, mariposite, and alurgite (terms which he thinks should be avoided). He also notes that the optical properties and specific gravity

cannot be correlated with chemical composition. The observations and con-
clusions of Schaller (1950) are in agreement with the results obtained at
this laboratory.

A few other investigators such as Whitmore, Berry, and Hawley
(1946); Hutton (1940); and Webb (1939) have discussed the relationships of
the above varieties or species to each other and to other members of the mica
family. However, very little structural work has been reported. Hendricks
and Jefferson (1939) note that uniaxial alurgite from St. Marcel, Italy,
has crystallized as the the 3-layer rhombohedral polymorph, whereas the so-
called alurgite from Cajon Pass, California, (Webb, 1939) has the normal mus-
covite structure. A study of the analyzed material from Cajon Pass has lead
us to the conclusion, in agreement with Schaller (1950), that it is merely
another muscovite and not alurgite. Whitmore, et al. (1946), in their work
on the chrome micas, note that mariposite conforms chemically with phengite,
which is in close agreement with Schaller, but regard it as a chromiferrous
variety of the mica. However they had no samples of mariposite suitable for
x-ray analysis and state that no suitable samples are recorded by other
investigators.

We have succeeded in obtaining additional specimens of high-silica
sericites, particularly a phengite analyzed by Pagliani (1937) and a mari-
posite described by Murdoch and Webb (1938). X-ray studies on these and
other specimens of high-silica muscovites have now been completed. The
range of the optic angle for the several varieties is shown below.

TABLE 4. VARIATIONS IN 2V OF HIGH-SILICA SERICITES

Name	2V	Source
alurgite	0-57°	Webb (1939, p.125)
mariposite	0-40°	Whitmore, et al. (1946, p.14)
phengite	0°-moderate	Kunitz (1924, p.383) and our observations

The fact that the 2V of these micas varies so much is important, because
those specimens that are nearly uniaxial invariably have crystallized as the
3-layer rhombohedral polymorph, whereas those that are markedly biaxial have
the normal muscovite structure.

Both uniaxial and biaxial alurgites from St. Marcel, the type lo-
cality, were x-rayed. Again, the uniaxial sections have crystallized as the
3-layer rhombohedral form and the biaxial portions as the 2-layer muscovite
form. The Penfield (1893) analysis, the only analysis of unquestioned

alurgite, apparently was made on material of both uniaxial and biaxial character. Penfield (1893, p.289) states:

> "The largest cleavage plates that have been observed are about
> 15 mm in diameter. When examined in convergent polarized light
> some show a uniaxial others a biaxial interference figure, the
> double refraction being rather strong and negative. ... The
> uniaxial character is undoubtedly the result of twinning, for
> plates can be found, which in some parts are uniaxial, in others
> biaxial, and where the plane of the optical axes is in two dif-
> ferent positions. Some, moreover, give a confused interference
> figure, as would be expected from two or more biaxial plates,
> placed one above the other in twin position. There is no dif-
> ference in specific gravity between the uniaxial and biaxial
> plates, they are identical in behavior before the blowpipe and
> there is every reason for believing that they are the same chem-
> ical substance."

Although it appears possible to simulate a uniaxial interference figure by systematically stacking thin muscovite sheets (Reusch 1869), it is structurally impossible to arrange the 2-layer muscovite structure in any way so that the 3-layer rhombohedral structure results.

Although Penfield (1893) states that alurgite is monoclinic, he notes (p. 289):

> "When treated with a mixture of strong hydrofluoric and sulphuric
> acids the biaxial plates show monosymmetric etching figures while
> those of the uniaxial plates are triangular or hexagonal, similar
> to those of muscovite and biotite, described by H. Baumhauer."

As was the case with Whitmore, et al. (1946), no mariposite suitable for single-crystal work could be obtained. However, several specimens of fine-grained mariposite (Nos. 996 and 1144) have been studied by the powder method. These specimens have the normal 2-layer muscovite pattern. Unfortunately, the material is so fine-grained and has such poor crystal development that it is impossible to determine the optical properties with certainty. It has definitely been observed, however, that the flakes ex-hibit considerable birefringence, thus indicating that the material is bi-axial. It is hoped that a sample of uniaxial mariposite described by Knopf (1929) may be obtained for x-ray study.

A specimen of the phengite analyzed and described by Pagliani (1937) has been received and studied (No. 1136). Pagliani reports that this material has a 2V of about 11°. However, the 2V of the specimen received is considerably larger than 11°, varying generally from 18° to 35°. Weis-senberg photographs of sections with 2V near 35° show the normal 2-layer

muscovite structure, whereas photographs of the sections with the smaller 2V have patterns of the 2-layered form combined with what appears to be the 3-layered rhombohedral polymorph. It is believed that patterns of the phengite with 2V of 11° (or preferably smaller) would indicate the 3-layered rhombohedral polymorph alone.

In Quarterly Report No. 3 is described a uniaxial muscovite, which crystallized with what we consider the first irrefutable 3-layer rhombohedral structure. It is also a high-silica mica; its chemical analysis by Jakob (1929; his No. 43) and showed 49.01% SiO_2, 3.91% MgO, and only 29.01% Al_2O_3. These percentages are generally characteristic of phengite. This mica probably was called muscovite by Jakob (1929) rather than phengite, because of its occurrence and paragenesis. The term phengite, it appears, has been used mainly for high-silica micas of secondary origin. Although this mica is predominantly uniaxial with the 3-layer rhombohedral structure, it was noted (Quarterly Report No. 3, p.3):

> "A few small areas in a few flakes show birefingence and have moderate 2V (approximately 40°). One such biaxial piece (1041b) that was isolated proved to have the normal 2-layered muscovite structure."

Again we have another example of the association of the 2-layer muscovite structure with the 3-layer rhombohedral type in a high-silica muscovite.

The mica from Mt. Mucrone described by Kunitz (1924) is also uniaxial and is called phengite by him. If specimens of this material could be x-rayed, we believe the structure would be that of the 3-layer rhombohedral polymorph. Attempts to obtain specimens of Kunitz's material have not been successful. The only mica approaching "normal" muscovite in composition and having a small 2V without an unusually high silica content is the 3-layer (so-called monoclinic) muscovite described by Axelrod and Grimaldi (1949).

Not all high-silica sericites, however, have small 2V and the 3-layer rhombohedral structure. In addition to the portions of alurgite, phengite, and mariposite mentioned above, Jakob's (1929) No. 44, a phengite, and Nos. 45 and 46, both sericites, have crystallized only as the normal muscovite polymorph (this is true at least of the portions of the samples furnished us for study). Jakob's (1929) Nos. 45 and 46, although they have the mica composition and powder photographs indicate the muscovite pattern, have very unusual megascopic appearances. The specimens are sericite schist fragments. It was thought possible that the high silica content of these specimens could be due to a small amount of admixed quartz in the analyzed material. Microscopic examinations revealed about 3-5% admixed epidote in No. 45, but No. 46 is essentially pure sericite.

There is clearly a definite relationship between formation of the 3-layer rhombohedral polymorph and high-silica "muscovite-like" micas. The only exception is the muscovite described by Axelrod and Grimaldi (1939). There is, of course, no certainty as to whether the few available analyses were carried out on uniaxial or biaxial material, but from the small-scale nature of the optical variation probably material of both types was involved in each analysis. The phengite of Pagliani (1937) is a good example, for the literature indicates the analysis was performed on material with a 2V of about 11°, whereas the material sent by Professor Pagliani establishes the fact that his phengite also has a pronounced biaxial phase. At first it was thought that the uniaxial portions might represent an approach to one of the octophyllite micas. However, this was ruled out, as the calculated atomic ratios from the available analyses indicate that all these micas are decidedly closer to the heptaphyllite (muscovite) group. It has been suggested by several authors that alurgite, probably because of its high MgO and FeO contents, is transitional between heptaphyllite and octophyllite micas. This hypothesis appears to be unacceptable: we believe that alurgite is a member of the muscovite-leucophyllite series. Why polymorphs as structurally different as the 2-layer muscovite and 3-layer rhombohedral forms occur together in the high-silica sericites remains to be answered.

FIELD WORK ON MICAS DURING JUNE-SEPTEMBER, 1952

During the period June-September, 1952, a moderate number of various micas were collected from widely scattered localities in the western part of the United States:

1. Muscovites, lepodilites, biotites, and rose muscovites (?) from several pegmatites in the Eight Mile Park District, Fremont County and the Guffey District, Park County, in Colorado.

2. Biotites from pegmatites and metamorphic rocks in Wind River Canyon, Wyoming.

3. Biotites from porphyritic hypabyssal alkalic intrusives, Bearpaw Mountains, Montana.

4. Muscovites from several pegmatites in the Petaca District, New Mexico.

5. A single small specimen of the very rare rose muscovite from the Globe pegmatite, Petaca District, New Mexico.

6. Chromiferrous muscovite, La Madera Mountain, Petaca District, New Mexico.

7. Rose muscovite and biotite from the Harding pegmatite, New Mexico.

8. Numerous samples of muscovite from several pegmatites in the Custer and Keystone Districts, Black Hills, South Dakota, including a suite of 10 muscovites and lithium-bearing micas from the well-zoned and well studied Hugo pegmatite near Keystone. This latter suite will afford a basis for a complete future paragenetic study.

SUMMARY OF INVESTIGATIONS, SEPTEMBER 1951-SEPTEMBER 1952

The preceding pages, in addition to discussing the results of the fourth quarter, also mark the completion of the first year of the natural mica studies. We believe that the results obtained thus far have outstripped our original estimates, and that future work expanded on the basis of this new information will prove equally fruitful. The quarterly reports, in general, contain a basic summary of the more important results obtained in each of the particular periods. Many data, however, particularly those concerned with optical and paragenetic studies, have barely been mentioned in these reports. These results will be incorporated in detail in the final report.

The primary findings of the natural mica studies during the first year may be summarized as follows:

1. There appears to be a distinct series between heptaphyllite muscovite and octophyllite lepidolite. "Lithium muscovite" is the name tentatively proposed for mica crystallizing with a hitherto unreported variation of the muscovite structure. It is considered to be approximately intermediate in composition and structure between muscovite and lepidolite and to represent the "missing link" in the series.

2. By means of the Weissenberg method polymorphic variations within individual sheets of lepidolite, as well as within books, have been found.

3. It appears possible to correlate the polymorphs in the muscovite-lepidolite series (except the uniaxial types) with Li_2O content. Tentative divisions are:

structure	Li_2O content
normal muscovite	0 - 3.0%
"lithium muscovite"	3.0 - 3.5%
"transitional"	3.5% - 3.8%
6-layer lepidolite	3.8 - 5.1%
1-layer lepidolite	above 5.1%

4. The 3-layer rhombohedral polymorph is similar to the 1-layer polymorph in composition. It is suggested that the 3-layer form may result from polysynthetic twinning of the 1-layer polymorph at 60° intervals, parallel with the base.

5. The physical development of micas in the muscovite-lepidolite series reflects in some measure their chemical positions in the series. Micas in the "transitional" position, for example, are characterized by small, imperfect crystals with anomalous optical properties.

6. Distinct and systematic variations in the optical properties of muscovites from different units within a pegmatite have been observed and measured. These variations represent basic differences in chemistry.

7. The rose muscovite problem has been studied completley, with the result that the paragenesis and significance of this variety are now completely understood.

8. As far as possible, the structures of the high-silica sericites or muscovites have been studied. However, this problem has not been completely solved.

9. Structural studies of the phlogopite-biotite series have not revealed any relationship between chemistry and polymorphism.

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