SYNTHETIC MICA INVESTIGATIONS, VI: *X*-RAY AND OPTICAL DATA ON SYNTHETIC FLUOR-PHLOGOPITE*

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ABSTRACT

Isomorphism studies in the synthetic fluor-mica group have shown the need for standard basic information on synthetic fluor-phlogopite ($KMg_3AlSi_3O_{10}F_2$).

Relatively large (up to 25 mm.), well-formed, single-crystal plates of synthetic fluor-phlogopite were obtained from one particular internal resistance melting experiment (method of synthesis described). Selected crystals were petrographically analyzed and found to be 99% pure. A chemical analysis was made.

Optical determinations gave the following values:

$\alpha = 1.522 \pm 0.001$ 2V = 14 ± 0.5°
$\beta = 1.548_5 \pm .0005$ (calculated) Biaxial negative
$\gamma = 1.549 \pm .001$

The following monoclinic unit cell constants were determined using a Geiger diffractometer:

$a_0 = 5.299 \pm 0.004$ Å Space Group = Cm (C_s^3)
$b_0 = 9.188 \pm .002$ Å $Z = 2$
$c_0 = 10.135 \pm .002$ Å $D_x = 2.879 \pm 0.004$ g/cm^3
$\beta = 99°\ 55' \pm 3'$

Using these values, all resolved maxima or significant doublets on an *x*-ray powder-diffraction pattern were indexed up to 77° 2*θ*, and an observed-calculated 2*θ* comparison showed good agreement. Single-crystal rotation and Weissenberg exposures were made. Optical goniometric measurements gave values in excellent agreement with those calculated from the unit cell data. The chemical and optical data were compared with literature values for both natural and/or synthetic material.

INTRODUCTION

The primary objective of the synthetic mica program at the Electrotechnical Laboratory† has been development of commercially practicable methods for manufacturing synthetic mica and fabricating mica products. Necessarily, the solution of engineering problems has received major emphasis; nevertheless, extensive investigations of a more fundamental character have also been made, of which this article represents one aspect.

One of the important requirements of a comprehensive mineral study is to identify precisely the compound chemically, optically, and struc-

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† This project has been conducted since 1947 in cooperation with the Office of Naval Research, the Bureau of Ships, and the Army Signal Corps.

turally. Precision measurements are presented herein on chemically analyzed, pure, single crystals of fluor-phlogopite which were produced in the internal electric resistance furnace. The data serve as bases of comparison for an extensive series of fluorine-mica isomorphs which have been prepared at this laboratory.

LITERATURE

Since an extensive review of the literature on synthetic mica is already available (1), only a brief sketch is presented here.

According to the older literature (2), synthetic fluorine-micas have been known for nearly 70 years; however the bulk of the work in this field has been done during the last 20 years in Russia, Germany, and Japan, and since the close of World War II, in the United States. The desire to produce synthetic mica as a commercial product, as indicated by early patents (3), has been a major stimulus to progress, although by no means the only incentive.

In the 10-year period 1934–44, Grigoriev, working on petrogenic and crystallochemical problems at the Leningrad School of Mines, published at least 18 papers describing his experiments on the synthesis of fluorine-micas and amphiboles. His work clearly established the equivalence of F^- and $(OH)^-$ in the mica structure and the practicability of crystallizing fluor-phlogopite ($KMg_3AlSi_3O_{10}F_2$) at atmospheric pressure from fluorosilicate melts. Although his early mica product (4) was a high-lime (6.43%) fluor-phlogopite (fluoride introduced as CaF_2), its x-ray diffraction pattern was very similar to that of natural phlogopite (5, 6). The refractive indices of a reasonably pure fluor-phlogopite made ten years later (7) were given as $\alpha=1.518$, $\gamma=1.554$.

Grigoriev's work probably was largely responsible for the renewed interest in the commercial development of synthetic mica. At any rate, in the mid 1930's, Siemens & Schuckert began an extensive engineering development to grow fluor-phlogopite in sheet form. By 1943 a pilot plant had been established, and a few crystals several inches in diameter were obtained by the slow crystallization of 100-kg. melts (8) contained in high-silica (82%) pots (9). Although these crystals were reported to be of high quality, no chemical, optical, or x-ray data on them are known to be available.

Concurrent with the Siemens development, an extensive crystallochemical investigation of fluor-phlogopite synthesis and isomorphism was conducted by Dietzel and Eitel (10, 11) at the Kaiser Wilhelm Institute for Silicate Research. Mica melts weighing about 150 g. were crystallized in carbon crucibles; the fluor-phlogopite crystals obtained were biaxial negative, with the principal optic plane parallel to (010);

$\gamma=1.545$, $2E=10°$ to $20°$. The similarity of their *x*-ray pattern to that of natural muscovite was noted but no data were given. Their attempts to synthesize fluor-muscovite ($KAl_2AlSi_3O_{10}F_2$) from melts of different kinds failed.*

Since about 1940, Japanese investigators (12) have contributed substantially to the literature on fluorine-micas, especially Noda and associates at the University of Nagoya (13), who have published at least 20 papers. Optical data were presented on fluor-phlogopite crystals from three representative experiments, as follows:

$\alpha=1.522$	1.544	1.513
$\beta=1.553$	1.564	1.539
$\gamma=1.554$	1.566	1.540
$2V=11°$	13°	14°
$c\wedge X=1.5°$	1.5°	0.5°

In another report (15), it was remarked that the refractive indices of fluor-phlogopite decreased as the amount of fluoride in the melt was increased beyond its theoretical content in the mica structure. However, no evidence was presented to show any change of fluoride content in the crystals.

The German development of synthetic mica terminated in 1945, but their accomplishments quickly became known to the world and prompted a series of similar mica investigations in the United States. First of these was a $1\frac{1}{2}$-year project (16) initiated in November 1945 at the Corning Glass Works in cooperation with Owens-Corning Fiberglas Corp., to check on the feasibility of producing synthetic mica commercially. The Bureau of Mines, Army-Navy cooperative engineering development program was an outgrowth of the Corning work. In 1946, an engineering program for the development of synthetic mica, sponsored by the Army Signal Corps, was started at the Colorado School of Mines (17). In the same year, exploratory experiments were started at the National Bureau of Standards (18), later to be continued as a basic research program sponsored by the Office of Naval Research. The Bureau of Standards measurements on fluor-phlogopite crystals probably are the most self-consistent and accurate presented to date (see Tables 1 and 2).

EXPERIMENTAL DATA

Synthesis of the Mica

The fluor-phlogopite crystals used in this study were prepared in a pilot-plant type of operation in which about 3 tons of mica batch were

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* Fluor-muscovite has been synthesized at the Electrotechnical Laboratory by solid state reaction of K_2SiF_6 with calcined kaolinite at temperatures of 500° to 700° C. It is unstable at higher temperatures.

melted and crystallized. The batch materials (technical grades) were compounded according to the following formula (27):

$$K_2SiF_6+6MgO+Al_2O_3+5SiO_2 \text{ (quartz)}$$
$$\text{Wt. }\%=25.5+28.0+11.8+34.7=100.0.$$

The major impurities were 1.5% CaO in the MgO and 0.7% Na_2O in the Al_2O_3, for which no compensations were made. It should be noted that the batch formula contains a 50 mol per cent excess of fluoride above the amount required in the theoretical fluor-phlogopite formula (F_6 instead of F_4).

About 15 tons of this batch were dry-mixed and loaded into a 12-foot high internal-electric-resistance melting furnace. The basic principle of operation of such a furnace (31) is illustrated by the accompanying sketch (Fig. 1). Melting was started at the center of the batch by means of an electrically heated graphite resistor, which was in contact with graphite electrodes. After several hours of operation, the resistor was consumed by oxidation and from then on the electric current was conducted through the melt. At all times the melt was contained, and the fluoride vapors were effectively sealed, by the surrounding mica batch. This particular melt (R-14) cooled under its own inertia at a rate of about 5° C./hour. Smaller melts have been cooled by gradual reduction of the electric power input (see ref. 31 for details).

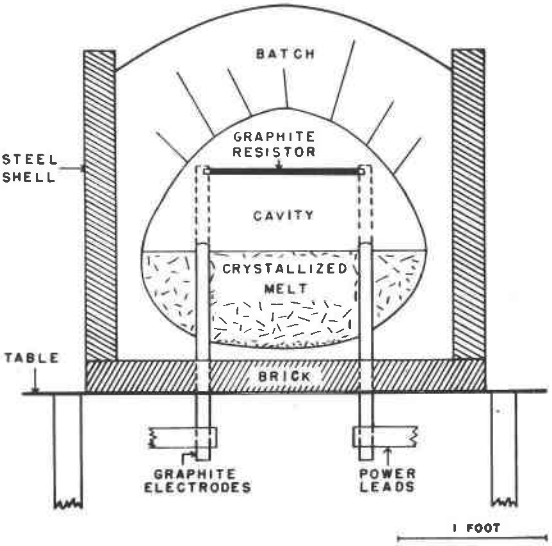

FIG. 1. Sectional view of model internal-electric-resistance melting furnace used for melting and crystallization studies of various synthetic mica batches.

The well-formed single-crystal plates selected for this study ($\frac{1}{4}$ to $\frac{3}{4}$ inch in diameter) came from a porous zone (crust) near the top of the melt. Simultaneous crystallization at the top and fusion at the bottom had resulted in the melt being slowly drained away from its crust, forming the porous stratum. In the massive state this material was slightly gray because microscopic particles of carbon were dispersed throughout the mass. As the carbon content did not exceed 0.013% by actual analysis, its effect on the properties investigated was considered negligible.

Chemical Composition

Several crystals were selected on the basis of freedom from inclusions and clarity. Optical examination of the crushed material showed the presence of 0.5 to 1.0% glass and a minute amount of MgF_2. Thus the sample was judged to be 99% pure. The results of a chemical analysis (No. 3689) are given in Table 1, which also includes an analysis (No. 3691) of selected pure crystals from the coarse zone above the middle of the melt (among the last to crystallize) to demonstrate the chemical uniformity of the fusion product. The table also includes an analysis by the Bureau of Standards (18) on its synthetic fluor-phlogopite.

With virtually no exception, chemical analyses of fluor-micas have consistently given low fluoride contents. In some cases this deficiency has been attributed to a substitution of oxygen for fluorine in the mica structure (4). The authors feel that no convincing evidence has yet been offered for this substitution in synthetic micas and would rather attribute the deficiency to errors in chemical analyses. The analytical procedure

TABLE 1. CHEMICAL ANALYSES OF SYNTHETIC FLUOR-PHLOGOPITE

Constituent	Theoretical Composition	No. 3689 Upper Porous Zone*	No. 3691 Coarse Middle Zone*	Bureau of Standards
SiO_2	42.79	42.50	42.70	41.87
Al_2O_3	12.10	12.35	12.21	12.97
Fe_2O_3	.00	.04	.06	—
MgO	28.71	28.36	28.58	28.27
CaO	.00	.14	.00	—
Na_2O	.00	.06	.06	.12
K_2O	11.18	10.88	10.92	10.94
F^-	9.02	9.30	9.20	8.52
O=F	−3.80	−3.92	−3.87	−3.51
Ignition loss (at 640° C.)	.00	.15	.20	—
	100.00	99.86	100.06	99.18

* Analysts: H. R. Shell and R. L. Craig.

developed and used in connection with the mica samples reported herein has recently been described by Shell and Craig (19).

The molecular formulas were calculated for the two analyzed fluor-phlogopites on the basis of 10 oxygen anions in the simplest possible formula weight, as follows:

No 3689—$(K_{0.98}Na_{0.01}Ca_{0.01})Mg_{2.99}(Al_{1.03}Si_{3.00})O_{10}F_{2.08}$
No. 3691—$(K_{0.98}Na_{0.01}Ca_{0.00})Mg_{3.00}(Al_{1.01}Si_{3.01})O_{10}F_{2.05}$

Thus it is evident that these micas closely approach the accepted theoretical composition for fluor-phlogopite, namely $KMg_3AlSi_3O_{10}F_2$.

Optical Properties

Refractive indices of the selected crystals were determined both with a petrographic microscope and an Abbe refractometer (method of grazing incidence) at 25° C., using sodium (D) light. Although the crystals measured had well-formed side pinacoids, they were not thick enough perpendicular to the basal face to give sharp lines as seen in the refractometer. Thus the accuracy of the refractometric determinations was reduced and judged to be about the same as that of the oil-immersion technique. α and γ were determined by both methods, and the values by each were found to be identical within the experimental error. Several relatively thick crystals from a previous experiment (R-7) were examined on the refractometer; their sharper field lines permitted greater accuracy, and values in agreement with those determined on crystals from the later experiment were obtained. The optical constants are listed in Table 2 and compared with those of a natural phlogopite (20) and with one synthesized at the Bureau of Standards (18). The table also includes the

TABLE 2. PHLOGOPITE OPTICAL CONSTANTS

	Authors' F-phlogopite*	West's Measurements	Bur. Standards F-phlogopite	Winchells' Phlogopite (20)
α	1.522 ± 0.001	1.5224 ± 0.0002	1.519 calc.	1.535
β	$1.548_5 \pm .0005$ calc.†	1.5489 calc.§	1.545	1.564
γ	$1.549 \pm .001$	$1.5494 \pm .0002$	1.547	1.565
2V	$14 \pm .5°$‡	14.4°	About 9°	About 10°
Optical Character	(−)	(−)	(−)	(−)

* M. V. Denny assisted in the optical determinations.

† The value for β was so close to that of γ that a calculation of the former was considered advisable.

‡ 2V was derived from measurements made on a 5-axis universal stage, using sodium (D) light.

§ West calculated $\gamma - \beta$ as 0.0005.

results of optical measurements made by West of Polaroid Corp.* on crystals synthesized at the Electrotechnical Laboratory in an earlier experiment (also R-7).

The substitution of fluoride for hydroxyl in the phlogopite structure obviously lowers the indices of refraction, as is the case with fluor-tremolite (21). The latter compound, however, shows virtually no change in 2V upon substitution, while the optic angle of fluor-phlogopite is noticeably larger.

X-ray Data

The powder-diffraction data for synthetic fluor-phlogopite were recorded, using a chart operation with the Philips high-angle *x*-ray diffractometer. The instrumental setting used was as follows: Scale factor, 16; multiplier, 1.0 (thus giving a counting rate of 800 counts per second, full scale); time constant, 4 seconds; Geiger overvoltage, 300 volts; divergence slit, 1°; receiving slit, 0.003 inch; scatter slit, 1°; scanning speed, $\frac{1}{4}$° per minute; chart scale, $\frac{1}{2}$ inch per degree; radiation, unfiltered Cu.

The sample subjected to *x*-ray analysis was obtained by mechanically shearing the previously-selected crystals. A minus 325-mesh portion was packed in the normal rectangular aluminum holder supplied with the high-angle diffractometer. The recorded maxima were read to 0.005° 2θ (0.0025 inch on the chart) by using a low-power binocular microscope; readings were " . . . taken at the midpoint of the peak at approximately two-thirds of the peak height . . . " (22). Instrumental corrections were applied from a smooth $\Delta\ 2\theta$ curve obtained by exposing a silicon standard compact (also supplied with the instrument) both before and after the fluor-phlogopite analysis. Corrections ranged from +0.030° at 77° 2θ to +0.070° in the low-angle region.

Since the monoclinic unit cell is defined by four parameters, it was necessary to establish accurately the positions of at least four clearly resolved and properly indexed peaks to solve the quadratic form (23). Such peaks were sought in the 2θ range above approximately 50°. Since mica usually gives an excellent $(00l)$ series, it was a fairly simple matter to determine the d_{001} dimension. The latter was derived from $(007)\alpha_1$† and $(008)\alpha_1$, both giving identical values of 9.983_6 Å. It remained to locate three additional maxima not included in the $(00l)$ series. The $(33\bar{1})\alpha_1$ maximum was determined at 60.450° by scanning the same region four times and averaging the resultant readings. A similar technique gave $(136)\alpha_1$ at 69.205°. No additional satisfactory peaks were available above 50° 2θ in the front-reflection region. It had previously been decided that

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* Section of status report No. 11 to the Office of Naval Research.

† $CuK\alpha_1$ taken as 1.54050 Å.

the back-reflection region was unsuitable owing to uncertainty in indexing. However, a back-reflection pattern* obtained from a relatively coarse sample (−100+200) was examined in the hope that preferred orientation might lend certainty to the indexing of an otherwise suitable peak. It was found that (139)α_1 could be indexed and, furthermore, that the nearby (0·0·10)α_1 peak could be used to determine the correction $\Delta\ 2\theta$. (139)α_1 was thus located at 101.825°, and enough data were available for solution of the quadratic form. These data are summarized in Table 3, along with the unit cell parameters derived therefrom.

Table 3. Monoclinic Cell Dimensions of Synthetic Fluor-phlogopite

Peak	2θ	Cell Dimensions (Angstroms)
$(33\bar{1})\alpha_1$	60.450°	$d_{100}=5.220\pm0.002$; $a_0=5.299\pm0.004$
$(136)\alpha_1$	69.205°	$d_{010}=9.188\pm\ .002$; $b_0=9.188\pm\ .002$
$(139)\alpha_1$	101.825°	$\beta=99°55'\pm3'=99.92°\pm.05°$
$(007)\alpha_1$	65.330°	$d_{001}=9.984\pm\ .001$; $c_0=10.135\pm.002$
$(008)\alpha_1$	76.225°	

Using the calculated cell dimensions and the space group symmetry, Cm (C_s^3), obtained from Weissenberg exposures, the positions of all permissible maxima in the 2θ range up to 77° (152 peaks) were calculated. Each resolved peak in this range was indexed; Table 4 compares the observed positions with those calculated from the cell dimensions. Only one maximum, (001)α ($\Delta\ 2\theta=-0.025°$), shows a deviation greater than 0.02°. The total of the positive deviations virtually cancels that of the negative differences, the actual unweighted average deviation being 2×10^{-4}°. The latter speaks favorably for the accuracy of the unit cell dimensions.

The literature was searched for accurate unit cell dimensions on either natural or synthetic phlogopite for comparison with the results of the present invesigation; however, no such data could be found.

The calculated density of synthetic fluor-phlogopite, on the basis of a bimolecular unit cell ($Z=2$), is 2.879 ± 0.004 g/cm.^{3} This compares favorably with the observed value of 2.88_2 g/cm.^{3}, determined† on a 77-mg. crystal with a Roller-Smith torsion microbalance (using toluene‡) at 25° C.

Rotation and Weissenberg (zero through third-level) exposures were made with rotation about the *a* axis, using unfiltered Cu radiation. The

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* Time constant, 8 seconds; divergence and scatter slits, 4°; receiving slit, 0.006 inch.

† Density determination by M. V. Denny.

‡ The crystal was soaked overnight in toluene, preparatory to the liquid weighing.

TABLE 4. X-RAY DIFFRACTION DATA (POWDER) FOR SYNTHETIC FLUOR-PHLOGOPITE
(SPACE GROUP *Cm*)

hkl	2θ obs.	2θ calc.*	$\Delta\ 2\theta$	Meas. Int.	d calc.
001	8.88°	8.855°	−0.025°	≫100	9.984 Å
002	17.765	17.77	+ .005	18	4.992
022	26.335	26.34	+ .005	7	3.380
003	26.765	26.765	0	≫100	3.328
112	28.48	28.465	− .015	9	3.133
$11\bar{3}$	30.80	30.82	+ .02	8	2.898
023	33.215	33.21	− .005	5	2.695
130	33.89	33.905	+ .015	4	2.642
$13\bar{1}$	34.315	34.32	+ .005	2	2.611
004	35.96	35.95	− .01	21	2.496
201	37.05	37.04	—	2	2.425
$13\bar{2}$		37.06			2.424
202	41.67	41.68	+ .01	2	2.165
$13\bar{3}$	41.72	41.725	+ .005	2	2.163
005	45.39	45.38	− .01	73	1.9967
134	52.535	52.545	+ .01	1	1.7402
$31\bar{1}$	52.73	52.725	− .005	1	1.7345
$13\bar{5}$	55.155	55.145	—	9	1.6641
006		55.15			1.6693
$33\bar{1}$	60.445	60.45	+ .005	6	1.5301
116	62.19	62.17	− .02	1	1.4918
205	63.265	63.265	0	1	1.4687
007	65.37	65.375	+ .005	6	1.4262
$11\bar{7}$	65.54	65.55	− .01	4	1.4229
027	68.86	68.87	+ .01	2	1.3621
136	69.24	69.245	+ .005	6	1.3557
$20\bar{7}$	69.385	69.38	− .005	4	1.3533
117	72.30	72.285	− .015	2	1.3059
206	72.34	72.345	+ .005	2	1.3050
008	76.225	76.225	0	7	1.2480

* Using λ CuKα (=1.5418 Å) below 25° 2θ and λ CuKα_1 (=1.54050 Å) above 25° 2θ.

rotation film led to a d_{100} value of 5.2 Å, whereas the zero-level Weissenberg gave d_{010} and d_{001} directly as 9.2 Å and 10.0 Å, respectively. None of the crystals *x*-rayed by single-crystal methods showed a structure other than Hendricks and Jefferson's (24), "single layer structure of the micas (monoclinic hemihedral)," the space group symmetry of which is *Cm*

(C_s^3). The single-crystal films also made possible several unambiguous indexings, on the basis of intensities, where two or more possibilities arose for a particular resolved maximum on the powder pattern.

Several selected crystals were measured on an optical goniometer. All of the crystals measured showed good to excellent basal pinacoids, side pinacoids, and hemi-prisms. Indications were seen of possible tetarto-bipyramid and hemi-side-dome faces. The goniometric data are summarized in Table 5.

TABLE 5. SYNTHETIC FLUOR-PHLOGOPITE GONIOMETRIC DATA

Angle	No. Times Observed	Range	Average	Calculated
(100)∧(110)	5	29° 35′–29° 37′	29° 36′	29° 36′
(001)∧(110)	6	81° 11′–81° 19′	81° 16′	81° 16′

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