

Biotite as a Primary Inclusion in Diamond: Its Nature and Significance

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Abstract

In recent mass spectrometric gas-analysis experiments involving the crushing of natural African diamonds that contained totally-enclosed mineral inclusions, a few small flakes of brown mica were recovered. They are identified as biotite with a Fe/(Fe + Mg) ratio of 0.61. Well-formed prismatic crystals of brown mica recovered from diamond-bearing kimberlite from Murfreesboro, Arkansas, have been found also to be biotite. The Fe/(Fe + Mg) ratio for the latter is 0.57. Chemical analyses, and optical and X-ray diffraction data are given for both biotites. Preliminary experimental data at 55 kbar suggest a similar biotite would decompose to garnet + liquid at about 1500 K. On the basis of the confirmed presence of biotite in diamond, of the decomposition products of biotite relative to pressure and temperature, and of related mineral and gas inclusions that have been identified in diamond, it is suggested that the stability boundary of iron-rich biotite may coincide closely with minimal growth conditions for natural diamond.

Introduction

Phlogopite is the usual mica associated with kimberlite, but there is only one very recent report of its ever having been found as an inclusion in diamond (Prinz *et al.*, 1973). There is one report in the literature of biotite included in diamond (Williams, 1932). Here, we confirm biotite as a primary inclusion in diamond.

In 1972, Melton, Salotti, and Giardini reported on included gas found within natural diamonds that contained primary mineral inclusions. This was determined by crushing externally-clean diamonds under a high vacuum in a chamber connected directly to the inlet of a mass spectrometer. Prior to crushing the diamonds were inspected under the microscope (45 \times) to insure that no detectable openings existed between internal inclusions and the outer surface of the diamonds. The gas released upon crushing contained water, carbon dioxide, methane, nitrogen, argon, and possibly hydrogen and carbon monoxide. Subsequently, Melton and Giardini have made additional analyses on diamonds from several localities, and have found a general persistence of included-gas in diamonds that contain solid inclusions.

Data from these additional tests are presented in a separate report (see Melton and Giardini, 1974), but from one test that involved a group of 39 small African diamonds each containing one or more

totally-enclosed inclusions, 3 flakes of brown mica ($\sim 0.1 \times 0.1$ mm) were recovered from the crushed sample. Prior to crushing, the diamonds had been treated with HF and boiling aqua regia, and then inspected at 45 \times to insure the absence of discernible cracks leading from the inclusions to the diamonds' external surface. On this basis, the inclusions are assumed to be of a primary nature; that is, as having been enclosed during growth of the diamond crystal. The gas released in the crushing experiment contained mostly CO and N₂, but also significant H₂O (9.6 vol. percent) and Ar (0.9 percent).

The recovered mica was subjected to microchemical, optical, and X-ray diffraction analyses, and was identified as biotite. For comparison, parallel analyses also have been carried out on two well-developed small crystals of brown mica obtained from a sample of diamond-bearing kimberlite from Murfreesboro, Arkansas. The analytical data show that this mica is also biotite, and almost identical to that recovered as a primary inclusion in African diamond. Other analyzed mica crystals from Arkansas kimberlite in which no diamond was found proved to be phlogopite.

Analytical data are given below. The significance of biotite in diamond is interpreted on the basis of these data, and high-pressure, high-temperature data on the stability of biotite.

Chemical Data

The chemical compositions of the mica samples were obtained with a Materials Analysis Company 400-S electron-probe microanalyzer. The unit was equipped with 3 X-ray spectrometers, one sealed and two gas-flow detectors, rubidium acid phthalate (RAP), PET, and LiF crystals. Instrument conditions were: acceleration voltage = 15 kV, specimen current = 0.5 microamps, beam diameter = 3 to 5 microns, spectrometer tuning = $K\alpha$, counting intervals = 20 seconds, except for Cr, F, and Cl. 100-second intervals were used for the latter. Due to the small size of the mica samples, they were mounted on a glass slide with epoxy glue and analyses were made on cleavage surfaces.

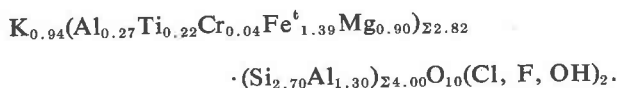
The analyses presented are averages from 20 to 30 spot counts. Reference standards consisted of two analyzed biotites (No. 1 and 3 from B. W. Evans) for all elements other than Cr. Orthopyroxene (No. 618-25.25, Univ. of Calif., Berkeley, collection) was used for chromium. The analyses of standards are available on request.

Corrections were made for background and drift. Since the standards were virtually identical to the diamond-related biotites, deadtime, adsorption, and fluorescence corrections were unnecessary. Accuracy is estimated to be within 1–2 percent, except for Cr_2O_3 (± 0.1 percent) and F (± 0.5 percent).

The chemical analyses of the respective biotites from within African diamond and from diamond-bearing Arkansas kimberlite are given in Table 1.

It was not possible to determine water contents, and the halogen content was not sought for the Arkansas crystals. The electron probe does not distinguish valence states, but the iron content of the biotites is assumed to be divalent and is so presented. This assumption is based on the coexistence of elemental carbon (diamond), a reducing gas (see Melton and Giardini, 1974), and the optical properties of the mica (see section on optical properties).

Table 1 reveals the chemical similarity of the two mica samples. The following ideal compositional formula for the biotite from the African diamonds is derived on the basis of 44 anionic charges after Foster (1960):



The chemistry of other mica crystals taken from Arkansas kimberlite in which no diamond was found are also included in Table 1. The absence of diamond is based on binocular microscope examination of heavy concentrates from the crushed rock. Near-colorless mica taken from dark-greenish-gray-colored coherent kimberlite, and brown mica taken from relatively friable green-colored kimberlite, were found to have iron contents of about 5 percent, considerably less than the approximately 20 percent content for the mica taken from kimberlite in which a small diamond was recovered. The diamond, by the way, was an elongated, rounded, colorless octa-

TABLE 1. Electron-Probe Chemical Analyses of Biotites from African Diamond and Arkansas Kimberlite

	Biotite				Brown mica from light-green kimberlite	Colorless mica from gray-green kimberlite
	recovered from African diamond		from diamond-bearing Arkansas kimberlite			
	Wt %	Cation ratio**	Wt %	Cation ratio***		
SiO_2	34.04	2.70	34.00	2.70	41.80	38.80
Al_2O_3	16.70	1.57	16.70	1.56	11.20	10.70
FeO	20.99	1.39	20.70	1.37	5.00	4.60
K_2O	9.33	0.94	9.25	0.94	9.80	0.30
MgO	7.63	0.90	8.90	1.05	23.50	31.20
TiO_2	3.63	0.22	3.20	0.19	6.10	4.30
Cr_2O_3	0.63	0.04	--	--	0.60	0.02
Cl	0.1	--	?	--	tr	0.01
F	1.50	--	?	--	--	--
Total	94.55		92.75		98.00	89.93

*Water was not determined. Iron is arbitrarily given as divalent (see text for explanation).
 **Fe/(Fe + Mg) = 0.61
 ***Fe/(Fe + Mg) = 0.57

hedron, about $0.03 \times 0.03 \times 0.09$ cm. It contained some included matter.

Optical Properties of Biotite

$2V$ is very close or equal to zero. The direction of α is approximately normal to the basal cleavage, and $\beta = \gamma = 1.66_8$. Birefringence is moderate, and a pleochroism in brown is noticeable. These properties apply to both iron-rich mica samples, and are in reasonable agreement with their chemistry. The chemical data show a presence of about 3 percent TiO_2 , and an approximate 2:1 abundance of MgO and TiO_2 . Heinrich (1946) has shown that titanium (and ferric iron) tend to increase γ , and that their influence is about twice that of divalent iron. The tendency of titanium to raise indices of refraction, therefore, would be counterbalanced by the magnesium content, and the mica would be expected to display optical properties consistent with ferrous biotite. The measured data indicate that the biotites in question are most closely classified as siderophyllite (Winchell and Winchell, 1951).

X-ray Diffraction Data

Initial X-ray diffraction work was done with the precession camera. The largest of the mica flakes recovered from the African diamonds was mounted on a glass fiber and oriented with (001) as close to perpendicular to the X-ray beam as possible. At 10° to the beam, a reciprocal lattice plane was found with one principal row parallel to the camera dial axis and another row normal to the X-ray beam. The row parallel to the dial axis was b ; assuming monoclinic symmetry, its repeat distance = 9.24_8 Å.

Subsequent to chemical and optical analyses, the mica flakes were crushed and a powder diffraction pattern taken (11.5 cm-diam. camera, $CuK\alpha$ radiation). In spite of fluorescence, a reasonable powder pattern (Table 2) was obtained. The pattern corresponds to those for biotite (ASTM-card 2-0045) and phlogopite (ASTM 10-0493).

The well-developed mica crystals from diamond-bearing Arkansas kimberlite were better suited for precession-camera analysis, and the following cell parameters were obtained: $a = 5.43_9$ Å, $b = 9.25_3$, $c = 10.23_7$; $\alpha = 90^\circ 10'$, $\beta = 100^\circ$, $\gamma = 90^\circ$.

After other analyses were completed, one of the Arkansas biotite crystals was crushed and a powder diffraction pattern taken. Data are included in Table 2.

TABLE 2. Powder Data for Two Diamond-Associated Biotites Compared to ASTM Data for Biotite and Phlogopite

Biotite* from diamond		Biotite** from kimberlite		Biotite† ASTM 2-0045		Phlogopite†† ASTM 10-0493		hkl
d (Å)	I/I ₀	d (Å)	I/I ₀	d (Å)	I/I ₀	d (Å)	I/I ₀	
10.11	100	10.11	100	10.1	100	10.13	100	002
-	-	5.00	5	-	-	5.056	20	004
-	-	4.63	20	4.59	20	4.612	20	110 020
-	-	4.39	5	-	-	4.515	5	021
-	-	-	-	-	-	4.079	5	112
-	-	-	-	-	-	3.814	20	023
-	-	3.550	5	-	-	3.540	30	114
3.362	60	3.350	90	3.37	100	3.362	100	006
-	-	3.282	20	-	-	3.283	40	114
-	-	3.155	5	3.16	20	3.156	5	115
3.026	50	3.052	50	-	-	3.040	40	025
-	-	-	-	2.92	20	2.926	5	115
2.838	20	2.819	10	-	-	2.818	20	116
-	-	2.661	10	2.66	80	2.651	20	131
2.631	10	2.629	80	-	-	2.624	100	116
2.521	10	2.518	20	2.52	40	2.522	30	008
2.440	20	2.442	40	2.45	80	2.439	40	133
-	-	-	-	-	-	2.361	10	117
2.305	10	2.309	5	-	-	2.304	5	220 040
-	-	2.275	10	2.28	20	2.270	5	135
2.180	10	2.181	40	2.18	80	2.180	40	135
-	-	-	-	-	-	2.039	20	224
-	-	2.015	30	-	-	2.017	60	0010
1.994	20	2.003	10	2.00	80	2.000	20	137
1.938	10	-	-	-	-	-	-	-
-	-	1.919	-	1.91	20	1.914	5	137
-	-	-	-	1.75	20	1.751	5	139
1.674	20	1.679	30	1.67	80	1.677	40	153
1.564	20	1.542	50	1.54	80	1.538	50	330 060
1.523	10	1.524	10	-	-	1.521	10	062
1.507	10	-	-	-	-	-	-	-
1.496	20	1.478	5	1.47	20	-	-	-
1.445	10	1.438	5	1.43	20	-	-	-
-	-	1.366	2	1.36	60	-	-	-
-	-	1.330	5	1.33	40	-	-	-
-	-	1.315	5	1.31	40	-	-	-

* biotite included within African diamond

** biotite from diamond-bearing Arkansas kimberlite

† 28.3% iron content

†† 2H₁ phlogopite

Experimental Results on Biotite Stability

We assume the biotite recovered from African diamond to be a primary inclusion since there is no evidence to the contrary. If correct, the biotite may be pre- or syngenetic to the diamond. The pressure and temperature conditions defining biotite stability, and the nature of its decomposition beyond these limits, may be of interest to the problem of natural-diamond genesis.

Eugster and Wones (1962), Wones and Eugster (1965), Markov *et al* (1966, 1968), Kushiro, Syono, and Akimoto (1967), and Yoder and Kushiro (1969) have explored the stability and decomposition products of biotitic mica relative to pressure, temperature, and H₂O. Modreski and Botchcher (1972) have similarly studied phlogopite + enstatite. Some limits of stability given by these workers are included in Figure 1.

Curve A of Figure 1 outlines the stability limit of annite given by Eugster and Wones for an oxygen fugacity of 10^{-22} . Decomposition products were sanidine, iron, and a vapor phase. Curve B delineates the incongruent melting of phlogopite under an hydrous gas phase to forsterite + liquid (Yoder and

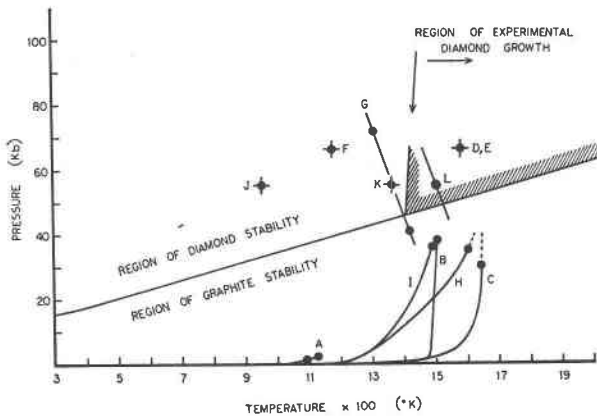


FIG. 1. Stability data on phlogopite, biotites, and annite relative to the graphite-diamond equilibrium boundary. Curve A represents the stability limit of annite under an oxygen fugacity of 10^{-20} (Eugster and Wones, 1962). Curves B and C are limits for phlogopite with and without water added, respectively (Yoder and Kushiro, 1969), and curve G, phlogopite without water added (Kushiro, Syono, and Akimoto, 1967). Curves H and I are stability limits for phlogopite + enstatite without and with added water (Modreski and Boettcher, 1972). Point D represents a point of initial decomposition for phlogopite, point E for phlogopitic biotite, and point F for lepidomelane according to Markov *et al* (1966, 1968). Points J and K are respective points of initial decomposition for annite (38.8 wt percent FeO) and biotite (25.5 wt percent FeO) according to experiments of this study. Curve L represents the approximated stability curve for biotite having 21 wt percent iron oxide (see text for details). Curve L would be expected to shift significantly to the left under a water-abundant environment.

Kushiro, 1969). Curve C is that given by Yoder and Kushiro for the breakdown of phlogopite to forsterite + liquid under gas-absent conditions.

Point D represents the experiment of Markov *et al* (1966) on phlogopite with 0.5 wt percent iron oxides. Decomposition products were pyrope garnet, an amphibole-like material, and two glass phases. Points E and F are similar experiments by Markov *et al* (1968) on a biotite with 9.9 wt percent iron oxides, and lepidomelane with 34.6 wt percent iron oxides, respectively. Garnet, an amphibole-like phase, and a glass phase were decomposition products. Curve G is drawn through the two experimental points given by Kushiro, Syono, and Akimoto (1967) for phlogopite having 13.1 wt percent iron oxides. Decomposition products were a pyrope-like garnet with about 5 wt percent K_2O , and an amphibole-like phase. Curves H and I are the respective boundaries of initial melting for phlogopite + enstatite with no water added (H), and with water added (I), given by Modreski and Boettcher (1972).

Eugster and Wones, Yoder and Kushiro, and Modreski and Boettcher worked below 40 kbar of confining pressure, and sealed their samples in platinum capsules. Under these conditions, the water content of the experiments was subject to control. Markov *et al* and Kushiro, Syono, and Akimoto, on the other hand, worked above 40 kbar, and contained their samples in graphite capsules. Water was not an added reactant.

The possible influence of graphite, loss of water of crystallization, and contamination from the solid pressure-transmitting material used in the pressure chamber upon the decomposition of mica are not known. It is not clear, therefore, whether the sets of data obtained below and above 40 kbar of confining pressure are comparable. To clarify this uncertainty, and also to seek data on biotite chemically close to that included in diamond, a number of experiments were carried out at 55 kbar with mica sealed in platinum capsules 0.3 cm-diam \times 1.6 cm long. The biotites used were annite from Rockport, Massachusetts (38.8 wt percent iron oxide), and mica from Yancey County, North Carolina (25.5 wt percent iron oxide). Chemical analyses are given in Table 3.

The mica was selected by hand-picking under a binocular microscope, washed with hot deionized distilled water, dried, and packed in cleaned platinum capsules. Eight 0.03 cm-thick Pt. disks were placed over the sample to act as a heat shunt during heli-arc welding of a press-fit cup-shaped platinum end-cap. The assembly was maintained at liquid-air temperature during welding. The sealed capsule was inserted into a thin-wall unfired pyrophyllite tube, and they in turn inserted into a tubular high-purity graphite furnace. Unfired pyrophyllite was used as a pressure-transmitting material in the pressure apparatus. Chromel-alumel thermocouples were positioned aside the hottest and coolest parts of the sample, and were read against ice. Corrections were applied for pressure effects according to the averaged values of Hanneman and Strong (1964) and Getting and Kennedy (1970). Pressure was established by room-temperature calibration of a similar set-up against transitions of bismuth (25.4 and 27 kbar), thallium (37 kbar), and barium (57 kbar).

The minimum temperature for annite decomposition at 55 kbar of confining pressure was found to be 960°K (J in Fig. 1). Residual phases found in the platinum capsule were biotite, almandine garnet,

TABLE 3. Electron-Probe Chemical Analyses of Initial Annite, Its Garnet Decomposition Product and Adjacent Altered Annite, and of Initial Biotite and Its Garnet Product

	Initial annite*	Garnet product**	Annite adjacent to the garnet**	Initial Yancey Co. biotite	Garnet product***
SiO ₂	35.6 wt %	39.2	42.2	36.2	38.7
Al ₂ O ₃	12.9	15.8	12.7	14.5	21.3
FeO	38.8	42.1	35.2	25.5	30.7
K ₂ O	9.0	0 to 0.2	5 to 10.5	9.3	0 to 1.4
MgO	0.05	tr	0 to 0.6	6.8	7.6
TiO ₂	2.3	0.4	0.2	2.8	0.3
Cr ₂ O ₃	0.05	0.2	0.3	0.03	tr
MnO	0.9	0.6 to 1.7	0 to 0.5	0.5	0.7 to 1.2
Cl	0.1	-	-	0.05	-
Total	99.7	99.0 av.	98.5 av.	95.7	100.2 av.

*from Rockport, Massachusetts.

**after 1100 K at 55 kbar for 20 minutes.

***after 1360 K at 55 kbar for 20 minutes.

and a glass phase. In contrast to Markov *et al* and Kushiro, Syono, and Akimoto, no amphibole-like phase was detected.

The garnet crystals were transparent orange-brown-colored dodecahedra and combination dodecahedra-trapezohedra. They commonly contained small black inclusions (possibly biotite) that showed high potassium and manganese contents. Potassium-rich inclusions might account for the unusual potassium content (~5 wt percent) reported by Kushiro, Syono, and Akimoto for their garnet. Electron-probe chemical analyses of the almandine garnet, and adjoining biotite crystals, respectively, are in Table 3. Analysis of the glass phase was not achieved, but a high potassium content is indicated.

In one experiment with annite, distilled water was added (15 wt percent). Extraneous thermocouple trouble prevented a good temperature reading in this test, but based on calibrated power input to the furnace and residual alteration of the surrounding pyrophyllite, a temperature less than 960 K yielded larger and more-perfectly developed garnet crystals than at 960 K without added water. Other differences were not noted. A lower temperature of decomposition in the presence of abundant water is indicated, and is very roughly estimated at \lesssim 900 K.

The biotite from Yancey County, N.C., showed initial decomposition at 1360 K and 55 kbar (K in Fig. 1). Similar decomposition products were produced, but the perfection of crystal development was inferior to that observed with annite. Chemical data on the garnet is included in Table 3, and X-ray diffraction data is given in Table 4.

Discrepancies apparently exist in reported temperatures and pressures of initial decomposition (see Fig. 1, especially G and K), and phases produced. However, the present experiments are adequate to show that the previously reviewed data on mica stability below and above 40 kbar are consistent with respect to olivine being a stable decomposition product below a pressure near 40 kbar, and garnet above. Furthermore, by assuming a reasonably linear relationship between iron content and decomposition temperature, the two values at 55 kbar (960 K for 38.8 wt percent FeO and 1360 K for 25.5 wt percent FeO) suggest a decomposition temperature close to 1500 K at 55 kbar (point L in Fig. 1) for a biotite with a 21 wt percent iron oxide content.

By projecting along a slope parallel to that given

TABLE 4. X-Ray Diffraction Data on the Garnet Decomposition Product from Yancey County, North Carolina, Biotite

d (Å)	I/I ₀ **	d (Å)	I/I ₀ **	d (Å)	I/I ₀ **
2.88	40	1.594	40	1.017	20
2.56	100	1.541	60	0.954	10
2.45	10	1.454	20	0.934	20
2.35	30	1.286	20	0.859	10
2.25	20	1.254	30	0.850	5
2.10	20	1.231	10	0.832	5
1.868	30	1.167	5	0.792	20
1.654	20	1.069	30	0.784	30
		1.051	20		

*subjected to 1360 K at 55 kbar for 20 minutes.

**visual estimates of relative intensity.

by Kushiro, Syono, and Akimoto (1967) for phlogopite (see curve G, Fig. 1), the stability curve for a biotite such as that found in diamond (21 wt percent FeO) shows intersection with the diamond-graphite equilibrium boundary at a point near 1500 K and 50 kbar. These values are also the minimum temperature and pressure for reproducible diamond synthesis in a metal-carbon system (Bundy *et al.*, 1961, Giardini and Tydings, 1962). This relationship is illustrated in Figure 1 (curve L).

Summary

The existence of iron-rich biotite as an inclusion in diamond is confirmed. It is also confirmed that biotite decomposes to olivine plus other phases below 40 kbar, and to garnet plus other phases above 40 kbar. Furthermore, olivine especially, and also garnet, are common inclusions in diamond (Meyer and Boyd, 1972). Both are common phases in kimberlite, as is mica. Melton and Giardini have shown that diamonds containing primary mineral inclusions yield upon crushing at about 500 K a gas phase whose average composition, in decreasing order of relative abundance, is H₂O, H₂, CO₂, CH₄, CO, N₂, Ar, and O₂ (based on data from 28 samples from Africa, Brazil, and U.S.A.; Melton and Giardini, 1974, plus data to be published).

The water and argon components of the gas may reflect a general chemical environment compatible with mica crystallization. So far, however, mica has appeared only rarely as an inclusion in diamond. The set of circumstances may indicate that biotite was only marginally stable under minimal conditions for diamond formation. The relative abundance of high-magnesium olivine and garnet inclusions, on the other hand, suggests that much diamond growth took place at temperatures beyond phlogopite stability, and at pressures below 40 kbar. If this speculation is correct, the only presently known mechanism for diamond formation compatible with such conditions is the decomposition of gases such as CO and CH₄ (Eversole, 1962). This type of mechanism is not inconsistent with the gas compositions that have been observed from natural diamonds.

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