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Some mineral inclusions from African and Brazilian diamonds: their nature and significance

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Abstract

Totally-enclosed inclusions of barite, biotite + chlorite, chlorite + biotite + garnet, chlorite + calcite, omphacite, pyrrhotite, and pyrrhotite + chalcopyrite from well-crystallized African diamonds, and forsterite, enstatite, and quartz from well-crystallized Brazilian diamonds, have been identified by X-ray diffraction and microchemical analyses. Totally-enclosed included matter known to date is reviewed and interpreted as indicating a broad range in chemical and physical environments during natural diamond formation. The range of included matter is consistent with the Harris-Middlemost evolutionary model for kimberlites, which proposes deep magmatic origin and interaction with country rock and successive fractionations during ascension.

Introduction

In spite of chemical, mineralogical, and petrological complexities of kimberlites, most students of diamond and kimberlite genesis have concluded that all natural diamonds formed at depths below 100 to 200 km under diamond-stable conditions defined by the graphite–diamond equilibrium boundary of the carbon system (Kennedy and Nordlie, 1968; Meyer and Boyd, 1972). However, the mechanism of natural diamond genesis is not yet understood. For example, it is not known whether diamond crystallized from a silicate melt independent of its chemical environment, under catalytic assistance such as practiced in commercial synthesis, or from a Fe–C–O–S system such as proposed by Marx (1972).

One obvious approach to the question of genesis is a detailed study of matter entrapped within diamonds. Such information, of course, also has relevance to the corollary question of kimberlite genesis. Meyer and Tsai (1976) attempted to join the two by applying the petrogenetic upper mantle model of Boyd (1973) to a select group of diamond inclusions, but results were inconclusive.

At present there are inadequate data to properly define *P*, *T*, and compositional boundary conditions. We give here new data on previously known inclusions of olivine, pyroxenes, mica, sulfides, and silica from African and Brazilian diamonds, and data on newly identified inclusions of barite, calcite, chalcopyrite, and chlorite from African diamonds. These and other foreign matter that have been found totally enclosed in natural diamonds seem consistent with the evolutionary model of kimberlite genesis proposed by Harris and Middlemost (1969), and suggest that diamonds formed over a broad span of kimberlite evolution.

Experimental method

Each inclusion was recovered from a different diamond. The twenty-three diamonds ranged from 0.25 to 0.5 carat, were colorless to light brown, well crystallized with octahedral, dodecahedral, and hexoctahedral forms, and were free of detectable surface cleavages at a magnification of 60×. Some Brazilian diamonds were obtained from Dr. Darcy Svisero, Sao Paulo University, Brazil. Other diamonds were purchased from commercial firms. Specific geographical origins are not known.

Inclusions were recovered by crushing diamonds in a piston-cylinder tool and picking through the fragments under a binocular microscope equipped with polarizers. Prior to crushing, diamonds were immersed in HF + 10 percent HNO_3 for 24 hours, with the first ten minutes under ultrasonic agitation. Crystals were then washed, boiled in aqua regia for 30 minutes subsequent to 5 minutes under ultrasonic agitation, then repeatedly washed with deionized distilled water.

Recovered transparent inclusions were examined with a petrographic microscope. All inclusions were examined with a scanning electron microscope, given a preliminary whole-inclusion X-ray diffraction analysis with an 11.46 cm diameter powder camera (filtered Cu radiation), remounted and polished for electron-probe chemical analysis, and finally recovered and crushed for powder X-ray diffraction analysis. Inclusions ranged from about 90 to 200 microns in cross dimension. Some were lost during attempted recovery from epoxy electron-probe mounts.

X-ray diffraction data are averages from several films, except where one or two superior patterns were obtained. Reference standards were used in the analyses.

Ten-element electron-probe microchemical analyses were conducted with a completely automated MAC 400S instrument. Operating conditions were 15 kV and 300 nA. Corrections were applied according to the methods of Bence and Albee (1968) and Albee and Ray (1970). Inclusions were mounted in epoxy, polished with diamond paste, then cleaned in ultrasonic baths. Tabulated data are averages from numerous spot analyses across polished surfaces and are believed to be accurate to ± 2 percent for major elements.

Experimental data: African diamond inclusions

The following mono- and polymineralic inclusions have been identified: (1) one of barite, (2) one of biotite + minor chlorite, (3) one of chlorite + minor biotite + minor garnet, (4) one of chlorite + minor calcite, (5) two of omphacite, (6) five of pyrrhotite, (7) one of pyrrhotite + minor chalcopyrite.

(1) Barite was recovered as a roughly equidimensional irregular red grain. Both SEM examination and X-ray diffraction analysis showed it to be finely polycrystalline. The diffraction pattern was essentially identical to that for synthetic barite. The chemical analysis (Table 1) shows that it is essentially pure $BaSO_4$, with only traces of Na, Ca, and Sr. This is the first known identification of barite as an inclusion in diamond.

(2) Biotite, with a minor amount of dispersed chlorite, was recovered as a dark reddish-brown elongated relatively euhedral crystal. Some porosity with numerous micaceous platelets existed adjacent to one edge. X-ray diffraction analysis showed the crystal to be mainly biotite but with a persistent presence of chlorite. The latter was indicated by very weak 7.21 A and 3.60 A reflections. The biotite diffraction data compare well with those given for 1M biotite. The bulk chemistry of the inclusion is given in Table 1. Low Al and K relative to Fe and Mg is qualitatively consistent with the presence of chlorite. Biotite has been identified previously as an inclusion in diamond by Williams (1932, Vol. 2, p. 420–426) and Giardini *et al.* (1974). But this is the first occurrence of biotite + chlorite, and suggests an early stage of hydrothermal alteration of the biotite at the time of its inclusion in diamond.

(3) Chlorite, with lesser quantities of biotite and garnet, plus a possible trace of clinopyroxene, were identified by X-ray diffraction in an elongated, rounded, dark green grain. Several diffraction patterns were obtained prior to chemical analysis. All showed relatively broad and weak reflections for chlorite and biotite, with chlorite the major phase. Garnet (a = 11.59 ± 0.02 A) was indicated by weak, spotty reflections, suggesting only one or a few grains. In addition, very weak 3.00 A and 1.62 reflections were also observed, and could indicate a possible presence of a small quantity of clinopyroxene. Bulk chemical data for the inclusion are given in Table 1. The garnet phase is probably not represented. Powder X-ray diffraction after chemical analysis indicated it was polished away in preparation for electron probe analysis. Chlorite was first inferred as an inclusion in diamond by Bauer and Spencer (1904), but this is the first definite identification. The minerals of this inclusion, and its rounded form, suggest an initial composition of biotite, garnet, and possibly pyroxene, with hydrothermal alteration of at least biotite prior to its inclusion in the host diamond.

(4) Chlorite, with a minor amount of nonhomogeneously distributed and surface-concentrated euhedral calcite crystals having preferred orientation, were identified in a green, approximately euhedral inclusion. Striations on one surface of the chlorite crystal appeared to be a cast of diamond-surface lineations. The whole inclusion yielded a powder diffraction pattern for chlorite showing orientation effects, plus a spotty pattern for calcite. The powder

Table 1. Average electron-probe chemical analyses of some inclusions from African diamonds

	Barite	Biotite with minor chlorite *	Chlorite with minor biotite, garnet & possible clinopyroxene **	Chlorite with minor calcite ***	Omphacite as octahedral cast	Omphacite, possibly twinned
SiO_2	-	40.1	37.3	34.3	53.9	53.1
MgO	-	22.4	27.6	40.3	8.6	15.0
FeO	-	21.2	11.3	9.2	7.5	8.2
MnO	-	0.7	0.5	-	0.6	0.6
CaO	tr	1.3	10.7	0.5	12.2	11.0
Na_2O	tr	0.5	-	tr	7.3	4.4
K_2O	-	4.6	1.7	tr	0.1	0.05
TiO_2	-	1.1	tr	tr	0.3	0.3
Al_2O_3	-	6.6	8.8	8.7	10.1	6.5
Cr_2O_3	-	-	tr	-	0.17	0.2
BaO	65.3	-	-	-	-	-
SO_3	34.7	-	-	-	-	-
SrO	tr	-	-	-	-	-
	100.0%	98.5%	97.9%	93.0%	100.8%	99.4%

* Bulk analysis. Total iron in this and all subsequent analyses is stated as FeO.

** Bulk analysis. Subsequent x-ray diffraction analysis showed that all or most of the garnet was lost from the inclusion during polishing for probe analysis. The identification of clinopyroxene is uncertain.

*** Electron-probe analysis performed only on calcite-free parts of the inclusion.

diffraction pattern yielded a pattern corresponding to 1b-type chlorite (Bailey, 1975). The chemistry of the calcite-free part of the inclusion (Table 1) shows it to be a magnesian penninite. This is the first identification of calcite as a syngenetic inclusion in diamond (Harris, 1968). Its existence conforms with the recognition of calcite as a primary magmatic mineral in kimberlite. The euhedral form of the inclusion, the diamond-like striations impressed on part of its surface, the localized surface presence of euhedral calcite crystals, and the combination chlorite + calcite, suggest an intense hydrothermal alteration in a CO_2-rich environment (Boettcher *et al.*, 1975) at the time of its inclusion.

(5) Omphacitic pyroxene inclusions were recovered from two diamonds. Both were sharply angular in form. One was a translucent green to yellow single crystal with an octahedral-like habit. The other inclusion was a transparent green fragment with sharply striated surfaces, possibly indicative of multiple twinning. Whole-inclusion X-ray diffraction patterns showed the octahedral-like specimen to be a single crystal, and that showing striations, a polycrystal. Powder diffraction data of both inclusions were essentially identical and in excellent agreement with ideal omphacite data given by Borg and Smith (1969). Chemical analyses of the two inclusions are given in Table 1. Omphacite included in African diamonds was reported by Prinz *et al.* (1975). An octahedral-like habit for pyroxene inclusions has been described by Sobolev *et al.* (1970) and Prinz *et al.* (1975).

(6) Bronze-yellow pyrrhotite inclusions were obtained from five diamonds. Three were round grains. Two were angular fragments of euhedral crystals. Except for the largest inclusion, X-ray diffraction patterns were weak and showed only the strongest pyrrhotite reflections. A good powder pattern from the largest specimen agreed with 4*C* monoclinic pyrrhotite. Interplanar *d*(102) spacings (based on the ideal NiAs structure) for all inclusions ranged from 2.06 to 2.07 A, indicating iron deficient compositions

(Arnold, 1956; Arnold and Reichen, 1959). Chemical analyses are given in Table 2. A sixth pyrrhotite inclusion from another diamond was found to contain a quantity of chalcopyrite. Its presence was initially indicated by 3.05 A and 1.86 A reflections, then confirmed by careful comparisons of the full diffraction data with standard patterns of chalcopyrite, pyrrhotite, and pentlandite. Powder diffraction data subsequent to electron probe analysis showed no chalcopyrite, indicating it was dispersed locally in the part of the inclusion lost in polishing for electron probe analysis. The chemical analysis reflects only pyrrhotite (Table 2). This is the first identification of chalcopyrite in diamond.

Experimental data: Brazilian diamond inclusions

The following monomineralic inclusions have been identified: (1) eight of forsterite, (2) two of enstatite, (3) one of quartz.

(1) Eight colorless forsterite inclusions from Brazilian diamonds were identified by X-ray diffraction. Some were euhedral and showed apparent pinacoidal, prismatic, and pyramidal forms. Others showed a more complex morphology, possibly similar to that described by Sobolev *et al.* (1970). Some were small irregular fragments, Two specimens were lost prior to chemical analysis. The best powder diffraction pattern from the largest specimem yielded unit-cell dimensions of a = 4.75, b = 10.26, c = 5.99 A. The chemical analyses of six inclusions are given in Table 3, and indicate a 93 ±1 percent forsteritic component. The compositions are consistent with analyses published by Meyer and Boyd (1972), Sobolev *et al.* (1972), Meyer and Svisero (1975), and Prinz *et al.* (1975), in that they are chromium-rich relative to olivine in kimberlite. It was noted, however, that the present inclusions showed a greater variation in chromium within individual inclusions (0.05–0.13%) than between the averaged values for respective inclusions (0.08–0.12%). The possible significance of chromium in diamond genesis has been discussed by Meyer (1975).

Table 2. Average electron-probe chemical analyses of five pyrrhotite and one pyrrhotite – chalcopyrite inclusion from African diamonds

	#1	#2	#3	#4	#5	#6 *
Fe	59.8	58.1	60.5	60.2	57.7	57.1
S	39.0	38.2	38.3	38.7	39.7	38.1
Ni	0.6	1.4	0.4	0.6	0.6	3.9
Cu	0.6	1.2	-	0.9	0.8	0.6
	100.0%	98.9%	99.2%	100.4%	98.8%	99.7%

* X-ray diffraction analysis subsequent to electron-probe analysis showed that the chalcopyrite content of the inclusion was lost during preparatory polishing.

Table 3. Average electron-probe chemical analyses of six forsterite inclusions from Brazilian diamonds

	#1	#2	#3	#4	#5	#6
SiO_2	40.87	40.91	40.78	40.97	41.07	40.80
MgO	51.45	51.90	50.32	51.75	50.74	50.78
FeO	6.58	6.26	7.91	6.47	7.30	7.35
MnO	0.19	0.19	0.12	0.10	0.17	0.19
NiO	0.25	0.21	0.14	0.17	0.18	0.17
Cr_2O_3	0.09	0.08	0.10	0.12	0.12	0.10
Al_2O_3	0.03	0.03	0.03	0.03	0.03	0.03
CaO	0.07	0.04	0.07	0.04	0.12	0.12
	99.53%	99.62%	99.47%	99.65%	99.73%	99.54%

(2) Two single-crystal inclusions of enstatite were identified by X-ray diffraction. Both were rounded transparent fragments with probable cleavage lamellae on fracture surfaces. One was light green, the other varied from yellow to green. Chemical analyses (Table 4) conform to enstatite. Similar inclusions have been described by Meyer and Boyd (1972), Meyer and Svisero (1975), and Prinz *et al.* (1975).

(3) One anhedral colorless inclusion of quartz was

Table 4. Average electron-probe chemical analyses of enstatite and quartz inclusions from Brazilian diamonds

	Enstatite #1	Enstatite #2	Quartz
SiO_2	57.27	57.16	99.9
MgO	36.10	35.85	-
FeO	4.47	4.57	-
MnO	0.09	0.12	-
NiO	0.20	0.23	-
CaO	0.27	0.19	0.01
Al_2O_3	0.80	0.77	0.01
Cr_2O_3	0.37	0.34	-
	99.57%	99.23%	99.92%

confirmed by X-ray diffraction and electron-probe analysis. It showed a lamellar texture with some pitting. Diffraction data showed it to be a single crystal in agreement with standard quartz to within ±0.002 A. Chemical analysis (Table 4) showed essentially pure SiO_2, with traces of Al and Ca. Quartz intergrowths parallel to (111) in Brazilian diamonds have been described by Colony (1923), Correns (1931), and Orlov (1959). Meyer and Svisero (1975) identified an euhedral quartz inclusion in a Brazilian diamond.

Discussion

Thus far, 29 minerals have been identified as totally-enclosed inclusions in diamond. They are diamond, graphite, forsterite, chrome pyrope, and pyrope–almandine garnets, enstatite, chrome–diopside, omphacite, kyanite, zircon, chromite, ilmenite, chrome–spinel, magnetite, rutile, coesite, quartz, pyrrhotite, pentlandite, chalcopyrite, pyrite, phlogopite, biotite, serpentine, chlorite, goethite, barite, calcite, and magnesite. Being totally enclosed within diamond, they are, according to Harris (1968), syngenetic with their host. We interpret inclusions which display sharp angular euhedral form, euhedral form with impressed diamond surface features, and diamond cast forms as syngenetic with their host diamond. Conversely, we interpret those inclusions with rounded forms as indicative of chemical solution or mechanical attrition prior to occlusion, therefore, pre-genetic to their host.

Crystalline inclusions in diamond have been found as dispersed crystals of one or more minerals, and as single inclusions of polymineralic composition. Examples of the latter are garnet + chrome diopside (Sobolev *et al.*, 1970), diopside + enstatite, and garnet + enstatite + olivine (Prinz *et al.*, 1975), biotite + chlorite, chlorite + biotite + garnet, and chlorite + calcite (this study).

In addition to visually-detectable solids, Fesq *et al.* (1975) have identified by neutron-activation analysis submicroscopic concentrations of O, Mg, Fe, Ca, Al, Na, and Ti, which they interpret as entrapped droplets of parental magma. Melton and Giardini (1976) gave mass-spectrometric evidence that the major impurity in diamond is oxygen.

Occluded volatiles have been identified in diamonds, both with and without detectable crystalline inclusions (Melton *et al.*, 1972; Melton and Giardini, 1974, 1975; Giardini and Melton, 1975a). The average composition from 36 diamond samples was, in decreasing order of abundance, H_2O, H_2, CO_2, N_2, CH_4, CO, CH_3CH_2OH, Ar, higher alkanes, and O_2 (Giardini and Melton, 1975b). The H_2O:CO_2 ratio varied among the 36 samples from about 15:1 to 1:2.

The common occurrence of euhedral, unaltered olivine inclusions with a high-chromium Fo_{93} composition, often with orientational and morphological influence by the host diamond (Mitchell and Giardini, 1953; Sobolev *et al.*, 1972; Prinz *et al.*, 1975), suggests these inclusions were closely syngenetic with their host. Their unique chemistry relative to the olivine of kimberlite (Meyer, 1975) suggests that these diamonds formed early in the evolution of kimberlite rock.

The occurrence of unaltered garnet inclusions, both euhedral (Harris, 1968), and xenohedral after diamond (Sobolev *et al.*, 1970; Prinz *et al.*, 1975), and their occurrence with olivine (Sobolev *et al.*, 1970; Prinz *et al.*, 1975) indicates that the garnet inclusions were also syngenetic with an early formation of diamond. But inclusions of chrome–pyrope and ultramafic minerals (*e.g.*, chromian diopside and olivine), and pyrope–almandine with eclogitic pyroxene, suggest that these diamonds formed over a more extended range of pressure and temperature.

Inclusions of unaltered biotite (Giardini *et al.*, 1974), and euhedral biotite with partial alteration to chlorite (this study) suggest that these diamonds formed at a still lesser depth, and in a H_2O-rich environment.

The chlorite + calcite inclusion (this study), with its euhedral crystal form, apparent surface impressions of diamond, and surface crystals of euhedral calcite, suggests that occlusion occurred in a highly fluid environment that was H_2O-rich during chloritization and CO_2-rich during calcite formation (Eggler, 1975; Boettcher *et al.*, 1975). Furthermore, field observations of calcite as a late-stage magmatic kimberlite mineral suggest that the host diamond may have formed at a relatively shallow depth.

The occurrence of coesite (Harris, 1968) and quartz (Meyer and Svisero, 1975; this study) as anhedral and euhedral inclusions suggests that these minerals were closely syngenetic with their hosts and that these diamonds formed under conditions roughly defined by the quartz–coesite equilibrium boundary (Boyd and England, 1960), under a water-rich environment (Eggler, 1975; Boettcher *et al.*, 1975).

The rounded and euhedral inclusions of pyrrhotite suggest that this sulfide formed before and during diamond formation.

The barite inclusion (this study) is consistent with the high concentration of barium observed by Fesq *et* *al.* (1975) in diamonds. It also is consistent with the model for kimberlite genesis proposed by Harris and Middlemost (1969). According to the model, the inclusion of barite suggests diamond formation at a late stage of kimberlite evolution.

In fact, of the models that have been proposed to explain what is known of kimberlites, the one by Harris and Middlemost is consistent with the range of foreign matter that has been found included in diamonds. The model proposes that kimberlites originated as ultramafic magmas at great depth beneath cratonic areas of the Earth. During ascension, in order to remain fluid, the magma underwent successive fractionations, together with enrichments of H_2O, CO_2, and incompatible residual elements such as K, Ba, Sr, *etc.* The concentration of major elements, and those which could substitute in their crystals, were functions of pressure, temperature, and the chemistry of the country rock at any particular depth. During the latter stages of evolution, incompatible elements such as K and Ba could become sufficiently concentrated to form specific minerals (*e.g.*, mica and barite).

Thus far, pristine high-chromium olivine, olivine with ultramafic minerals (*e.g.*, enstatite), eclogitic minerals (*e.g.*, omphacite), hydrous silicates (*e.g.*, biotite and chlorite), coesite, quartz, calcite, barite, and water-rich volatiles have been found totally enclosed in natural diamonds. A plausible interpretation of these occluded impurities is that natural diamonds formed over a broad range of chemical and physical environments such as described in the Harris-Middlemost model for kimberlite genesis. In spite of this interesting apparent correlation, however, Nature's mechanism (or mechanisms) of diamond formation remains unsolved.

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