

## Microinclusions in Dark Gray Diamond Crystals of Octahedral Habit from Yakutian Kimberlites

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**Abstract**—Micrometer-sized inclusions in dark gray diamond crystals of octahedral habit from Yakutian kimberlites were studied by analytical scanning electron microscopy and analytical transmission electron microscopy. In addition to peridotite-suite silicate inclusions (enstatite and olivine), which were previously studied in detail as macroinclusions in perfect diamond crystals, there are abundant inclusions of native metals (Fe–Cr and Fe–Cr–Ti intermetallic compounds and metallic Fe), sulfides (Fe–Ni sulfide, polydymite, and chalcopyrite), and carbonates (calcite). Rare grains of ilmenite, fluorite, apatite, zircon, phlogopite, and some other minerals were also found. The gray and black colors of diamonds were previously attributed to the presence of graphite. Although this phase was identified in the electron diffraction patterns of all the samples, its content was very low. Microinclusions of induced octahedral morphology composed of intimate enstatite–calcite intergrowths were also found. Possible deep processes responsible for the formation of diamonds with such a peculiar inclusion association are discussed in light of new data on experimental synthesis.

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### INTRODUCTION

Investigations of mineral inclusions in natural diamonds are of special interest as characterizing the crystallization medium of the diamonds and providing information on the processes of deep mineral formation in general. Up to date, a number of studies have been published [1, 2] on the composition and structure of mineral inclusions in diamonds. Based on these data, it was concluded that diamonds crystallized in silicate mantle rocks, peridotites and eclogites.

However, these studies focused mainly on relatively large inclusions in perfect transparent crystals. Such inclusions can be identified by means of optical microscopy and, after polishing of the host crystals, studied by electron microprobe analysis and other methods. On the other hand, micrometer- and submicrometer-sized inclusions, which are abundant in distorted semitransparent diamond crystals, have long remained poorly investigated. Recent studies of distorted diamonds of cubic habit, diamonds surrounded by opaque coats [2–5], and transparent octahedral crystals with cloudy zones [6] revealed unusual inclusions in them. In particular, numerous melt or fluid microinclusions of complex carbonate compositions and microinclusions of carbonates, micas, and some other minerals were found in the diamonds of cubic habit and coated diamonds. In

addition to silicates of the eclogitic and peridotitic parageneses, microinclusions of carbonates, phlogopite, and brines containing Cl, K, carbonate, and water were detected in the diamonds with cloudy zones.

The goal of this study was to investigate microinclusions in semitransparent dark gray diamonds of octahedral habit from Yakutian kimberlites using analytical scanning electron microscopy (ASEM) and analytical transmission electron microscopy (ATEM).

It is known that diamond crystals of octahedral habit and products of their partial dissolution of rhombododecahedral habit are most widespread in almost all deposits of the world. Semitransparent and dark gray crystals containing numerous microinclusions are widespread among them. For instance, their content in some kimberlite pipes of Yakutia is up to 40% [7]. Black inclusions in them are usually identified by visual inspection as graphite. In this connection, it is interesting to investigate microinclusions in dark gray diamonds of octahedral habit using modern methods of analytical electron microscopy.

The methods of analytical electron microscopy combining scanning and transmission electron microscopy and energy-dispersive chemical analysis are especially efficient for the investigation of micrometer-sized

inclusions in opaque diamonds, because they do not require their preliminary visual detection.

Using this combination of methods, we previously investigated inclusions in opaque cryptocrystalline diamond aggregates from Brazilian and Central African placers and Yakutian kimberlites [8–10], fine-grained diamond aggregates from the kimberlites of China [11], coarse-grained aggregates from Yakutian kimberlites [12], semitransparent crystals of cubic habit from the placers and kimberlites of China [13–14], and semitransparent rounded crystals from Australian lamproites [15]. This work revealed a great diversity of mineral inclusions in natural diamonds. One of the most intriguing results was the demonstration of the widespread occurrence of native metal inclusions: Fe, Cr, Ni, Ag, and others, in natural distorted diamonds. In addition to native metals, inclusions of oxides (magnetite, ilmenite, etc.), sulfides, carbonates (calcite, dolomite, etc.), chlorides (halite and sylvite), and many other unusual minerals were found in these diamonds. Silicate inclusions of the peridotitic and eclogitic parageneses were not found in the cryptocrystalline diamond aggregates and occur as rare grains in the fine-grained and coarse-grained aggregates.

## SAMPLES AND METHODS

Four diamond crystals of octahedral habit weighing 1.5–2.0 carats from Yakutian deposits were studied. The crystals are semitransparent and have a dark gray color owing to the presence of numerous microinclusions. They were selected from a large number of similar crystals on the basis of the absence of deep cracks and etch channels. Crystal no. 1 is the darkest and almost black, and crystal nos. 3 and 4 show a brown tint. Sample no. 1 showed a parallel striation and polycentric development of faces; sample no. 2 showed a fine parallel splintery striation; and sample nos. 3 and 4 exhibited splintery striation and a droplike sculpture on dissolution surfaces. All the crystals are significantly distorted. Mechanical cracks were observed in sample nos. 3 and 4.

Before the investigations, each diamond crystal was crushed using a small metal anvil, taking care to prevent sample contamination with metal particles. To this purpose, a diamond sample was wrapped in several sheets of thick paper. The fresh fractures of the largest fragments were then coated with carbon for charge stabilization and studied in detail using a JSM-5300 electron microscope (JEOL, Japan), equipped with a Link-ISIS energy-dispersive spectrometer (Oxford, England). To increase the accuracy of chemical analyses, some fragments were additionally ground and reanalyzed on an analytical scanning electron microscope. Using the Link-ISIS energy-dispersive analyzer, the concentrations of chemical elements from Be to U, including O, can be determined semiquantitatively in fresh fractures and quantitatively in polished surfaces.

A finer (dustlike) fraction produced during the crushing procedure was loaded into an ampoule with distilled water and further crushed for 7–10 min mechanically and using an UZDN-A ultrasonic disperser. Then, several droplets of the produced suspension were applied onto a special copper grid coated with an electron-transparent carbon film. After drying, the specimens were investigated using a JEM-100C transmission electron microscope (JEOL, Japan), equipped with a KeveX-5100 energy-dispersive analyzer (USA) for the qualitative analysis of elements from Na to U.

Thus, the analytical procedure provided comprehensive information on micrometer-sized and submicrometer inclusions in diamonds, some of which remained on fracture surfaces and some were transported into suspension specimens.

## RESULTS

### *Analytical Scanning Electron Microscopy*

Mineral inclusions on the surfaces of fresh fractures produced by the crushing of diamond crystals were investigated by ASEM. In addition, some fracture surfaces were ground and polished for further investigation and more accurate analysis. The following inclusions were identified and analyzed on the fresh fractures.

The maximum number of inclusions was detected in sample no. 1. The most common among them are Fe–Cr intermetallic compounds, enstatite, calcite, Fe<sub>2</sub>NiS<sub>4</sub>, and polydymite. In addition, rare grains of the Cr<sub>0.58</sub>Fe<sub>0.39</sub>Ti<sub>0.03</sub> intermetallic compound, ilmenite, and monticellite were found.

Inclusions of Fe–Cr intermetallic compounds, calcite, and enstatite were identified in sample no. 2.

Inclusions of Fe–Cr intermetallic compounds, calcite, Fe<sub>2</sub>NiS<sub>4</sub>, pyrite, and olivine were found in sample no. 3.

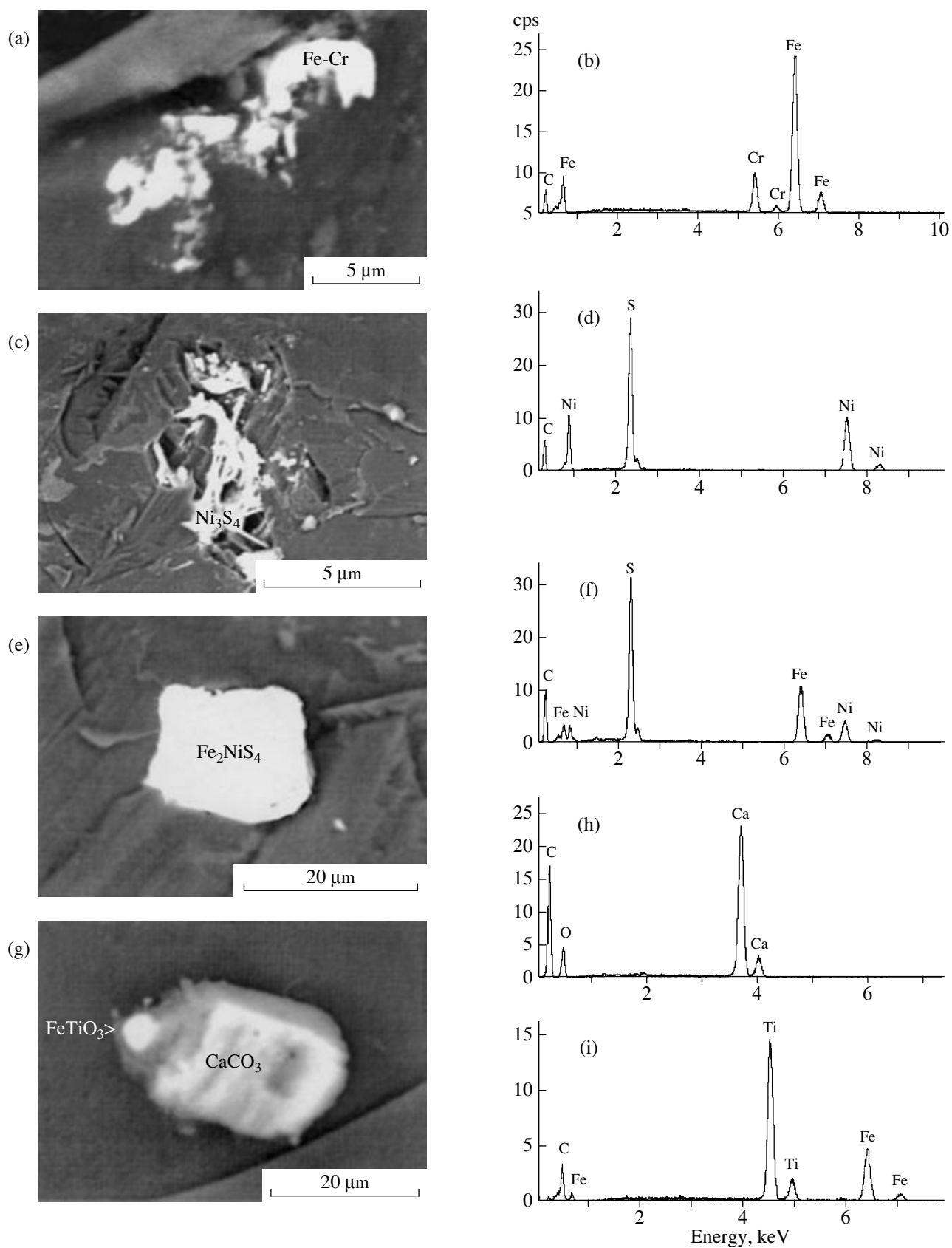
Numerous inclusions of Fe–Cr intermetallic compounds were only observed in sample no. 4.

The following microinclusions were detected on the polished surfaces of diamonds: Fe–Cr intermetallic compounds, enstatite, and olivine in sample no. 1; enstatite in sample no. 2; and lawsonite in sample no. 3.

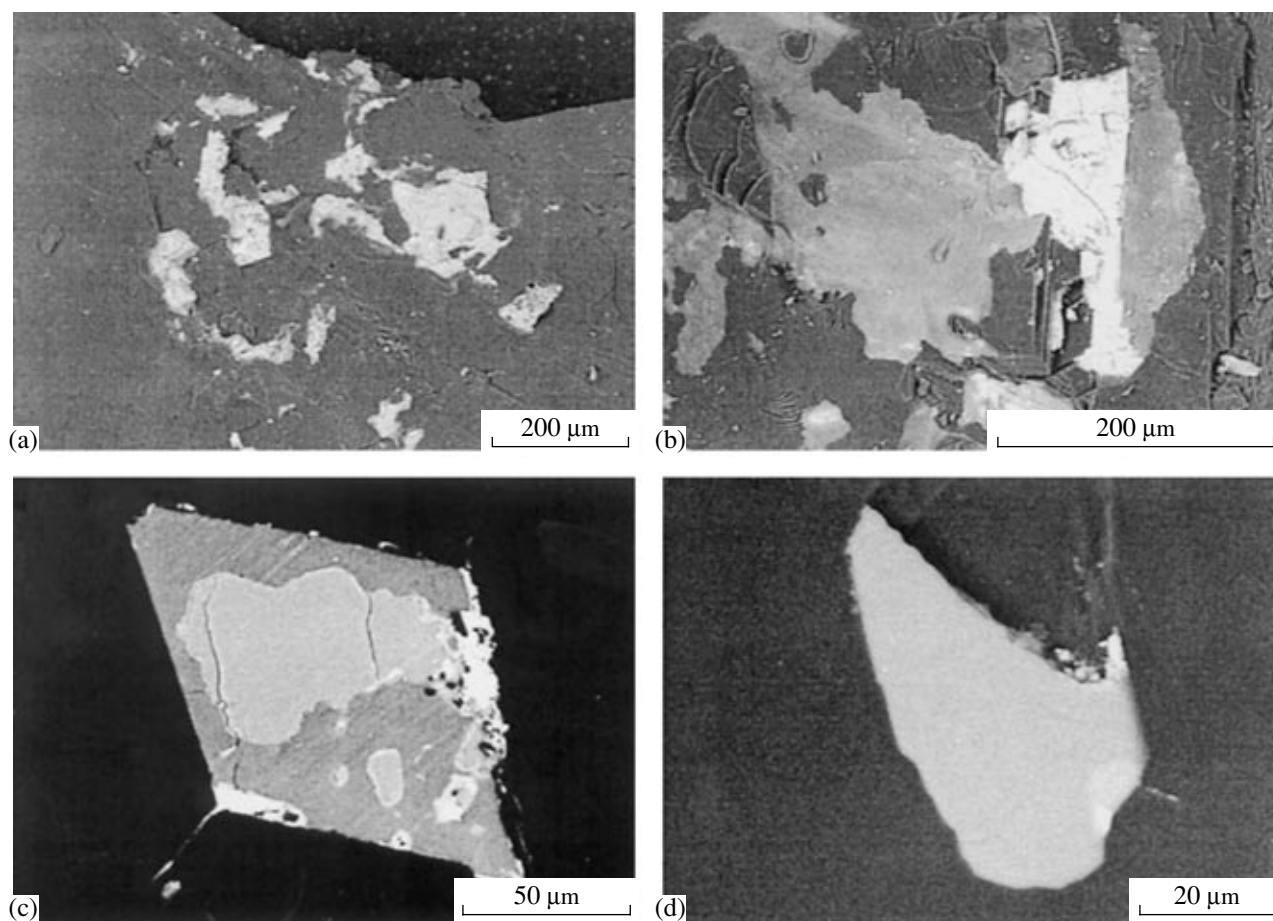
Consider some mineral phases detected in the diamonds by means of ASEM.

**Fe–Cr intermetallic compounds** were most frequently observed on the fractures of all the diamond crystals as isometric and elongated particles or aggregates, varying in size from less than 1 μm to 10 μm (Figs. 1a, 1b). Their composition is dominated by iron, the content of chromium is 11.0–13.4 wt %. The intermetallic compounds usually contained minor admixtures of Ni, Co, Mn, Ti, and Cu (less than 1 wt %).

**Cr<sub>0.58</sub>Fe<sub>0.39</sub>Ti<sub>0.03</sub> intermetallic compound** was observed in sample no. 1 as an isometric particle, about



**Fig. 1.** Backscattered electron images and energy-dispersive spectra of microinclusions in the diamonds: (a, b) Fe–Cr intermetallic compounds; (c, d) polydymite; (e, f)  $\text{Fe}_2\text{NiS}_4$ ; and (g–i) calcite–ilmenite intergrowth.



**Fig. 2.** Backscattered electron images of silicate inclusions on (a, b) fresh fractures and (c, d) polished surfaces of diamonds: (a) aggregate of enstatite crystals; (b) intergrowth of enstatite particles approaching the ideal formula (bright) and particles with excess Mg (gray); (c) intergrowth of enstatite (dark gray) and calcite (light gray), the octahedral shape is induced by the host diamond; and (d) olivine grain. Bright particles at the boundary of the enstatite–calcite inclusion (c) are abrasive grains.

10  $\mu\text{m}$  across. Its composition corresponded to the formula with minor admixtures of Zn (0.52 wt %) and Ni (0.51 wt %).

**Polydymite** was represented by elongated aggregates of isometric particles (Figs. 1c, 1d) and, occasionally, individual particles. Its composition approached the ideal formula  $\text{Ni}_3\text{S}_4$ . Some particles contained Fe and Cr impurities.

**$\text{Fe}_2\text{NiS}_4$ .** Several particles of this sulfide were found. They showed no distinct geometrical shapes. Only some particles of this mineral had roughly rectangular outlines (Figs. 1e, 1f). The composition of this mineral is close to the formula  $\text{Fe}_2\text{NiS}_4$ . Some particles contained admixtures of Cr (up to 0.21 wt %), Co (up to 0.69 wt %), and Cu (up to 0.69 wt %). There is no mineral with such a formula in handbooks. It is possible that these inclusions are a new mineral. The mineral violarite contains the same elements but in different proportions:  $\text{FeNi}_2\text{S}_4$  [16].

**Calcite** crystals are rather common and form inclusions of variable shapes and sizes. Both monocrystalline particles with clear linear boundaries, tens of

micrometers in size, and isometric rounded grains, up to 150  $\mu\text{m}$  in size, were observed. Polymineralic inclusions formed by intimate intergrowths of calcite and ilmenite (Fig. 1g), polydymite, and enstatite were found (Fig. 2c).

**Enstatite** was observed as relatively large irregular grains, up to 200  $\mu\text{m}$  across, which were in intimate intergrowth with the diamond matrix. Such inclusions were especially abundant in sample no. 1, where they occasionally accumulated as separate groups (Fig. 2a). Individual irregular enstatite grains, 10–15  $\mu\text{m}$  across, are less common. Of special interest was an inclusion observed on the polished surface of sample no. 2. This inclusion had an octahedral shape with a longer diagonal of 170  $\mu\text{m}$  (Fig. 2c). This is a typical inclusion the shape of which is induced by the host diamond. The mineral material of the inclusion is close to enstatite in composition, and rounded grains of pure calcite were observed in its central part.

The chemical analysis of fracture surfaces in the diamonds showed that inclusions having a composition approaching the theoretical enstatite formula associ-

ated with grains with an excess of Mg and Fe (up to 30.17 wt % in total) and a deficit of Si (up to 23.39 wt %). A distinct boundary was observed in some grains between chemically normal enstatite and enstatite with an excess of Mg and Fe and a deficit of Si (Fig. 2b). In order to minimize the analytical error related to the roughness of the surface, some diamond chips were polished and reanalyzed. However, practically the same results were obtained on polished surfaces: in addition to inclusions of enstatite with almost ideal compositions, there were grains enriched in Mg and Fe at the expense of Si. One possible explanation for the observed departure of the composition of some enstatite inclusions from the ideal formula is that they are microscopically heterogeneous and partly replaced by chrysotile; tiny particles of the latter mineral, less than one micrometer in size, were detected during the investigation of these diamond samples by ATEM.

**Olivine** corresponding in composition to forsterite was found as an elongated irregular grain about 65  $\mu\text{m}$  in size (Fig. 2d) and as an octahedron with a longer diagonal of about 25  $\mu\text{m}$ , the formation of which was induced by the host diamond.

#### *Analytical Transmission Electron Microscopy*

Using the ATEM method, inclusions in the diamond crystals of octahedral habit were identified from their chemical composition, and their structural characteristics were determined from the analysis of electron diffraction patterns.

Inclusions in sample no. 1 were represented by Fe–Cr intermetallic compound, graphite, chalcopyrite, ilmenite, rutile, fluorite, apatite, calcite, and clinochrysotile.

Inclusions in sample no. 2 are Fe–Cr intermetallic compound, metallic iron ( $\alpha\text{-Fe}$ ), graphite, ilmenite, rutile, calcite, apatite, zircon, and phlogopite.

Inclusions in sample no. 3 are graphite, calcite, fluorite, Ca–Mg silicate, and clinochrysotile.

Inclusions in sample no. 4 are graphite, Ca–Mg silicate, and clinochrysotile.

Consider the characteristic features of some of these inclusions.

In the suspension specimens produced from the diamond samples, pure metallic iron ( $\alpha\text{-Fe}$ ) and iron with a small admixture of chromium (Fe–Cr intermetallic compound) were observed as irregular particles with rounded outlines and thinned margins (Fig. 3a). They ranged in size from less than one micrometer to a few micrometers. The electron diffraction patterns of such particles (Fig. 3b) were always ring-shaped, which is typical of polycrystalline aggregates, and displayed several intense reflections of the  $\alpha\text{-iron}$  structure: 111, 220, 311, etc.

Graphite was observed both as normal scales with typical electron diffraction patterns with well-defined spots and as rounded globular particles of variable thickness, up to 2  $\mu\text{m}$  in size (Fig. 3e). Structurally, the

latter are polycrystalline aggregates with distinct ring electron microdiffraction patterns. The electron microdiffraction pattern shown in Fig. 3f consists of the main reflections of the graphite structure, (100) and (110), and spatial reflections observed owing to a small tilt of the sample.

Chalcopyrite occurs as compact aggregates, up to 1  $\mu\text{m}$  in size (Fig. 3c). They show discrete ring microdiffraction patterns (Fig. 3d) indicating a preferential orientation of some microblocks within the particles.

Calcite was represented in the suspension specimens by massive angular fragments and flattened distorted microcrystals, the electron microscopic images of which showed characteristic interferential fringes (“moir”) indicating a high degree of crystal perfection (Fig. 3g). The electron microdiffraction patterns of calcite particles always consisted of well-defined spots, which is characteristic of monocystals (Fig. 3h).

Fluorite occurred as polycrystalline aggregates, in which individual crystals, tenths to hundredths of micrometer in size, could be discerned, and as individual monocystals often altered and etched along margins.

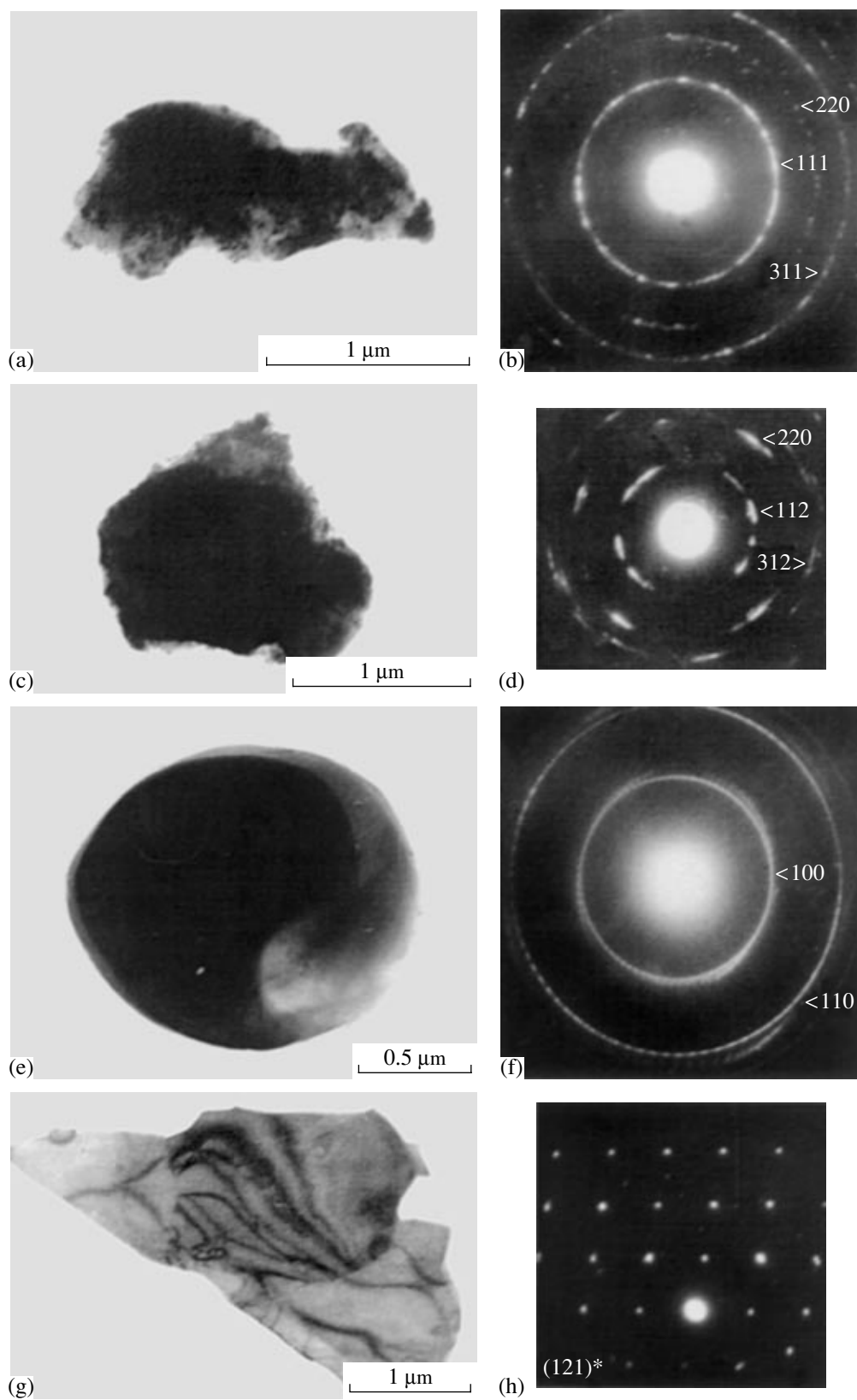
Chrysotile did not form usual elongate thin fibers but occurred as peculiar compact bundles (forming columns). The analysis of their electron microdiffraction patterns allowed us to identify the mineral as clinochrysotile. In addition, the microdiffraction patterns suggested that the layers in such bundles are parallel and there is no disorientation.

The Mg–Ca silicate was detected from the presence of Si, Mg, and Ca in its energy-dispersive spectra and was represented by isometric particles, less than one micrometer in size. More detailed phase identification was hampered by the ambiguous interpretation of its diffraction patterns.

## DISCUSSION

Our investigations of micrometer-sized inclusions in the dark gray diamonds of octahedral habit revealed the presence of peridotite-suite silicate inclusions of olivine and enstatite, which have previously been detected among macroinclusions in octahedral diamond crystals from various deposits over the world [1]. It should be noted that they were rather common in three of the four samples, and enstatite microinclusions even formed three-dimensional clusters in sample no. 1, which was never previously reported in the literature. The observed deviations of the compositions of some enstatite inclusions from the ideal formula were described previously in distorted diamonds of cubic habit from the kimberlites of Botswana [3].

The octahedral polymineralic enstatite–calcite inclusion is of special interest. The formation of its octahedral shape is related to the inducing influence of the host diamond, the force of crystallization of which is known to be higher than that of other minerals. Mac-



**Fig. 3.** Transmission electron microscope images and microdiffraction patterns of inclusions in the diamonds: (a, b) metallic Fe; (c, d) chalcopyrite; (e, f) graphite; and (g, h) calcite.

roscopically visible inclusions of enstatite and other minerals with induced octahedral morphologies were previously described in crystals of octahedral habit from Yakutian deposits [17]. Syngenetic inclusions of intergrown pyroxenes and Ca and Mg carbonates were recently found in diamonds from Namibian placers [18]. In addition, micrometer-sized inclusions having a bulk carbonatite composition were investigated in distorted diamonds of cubic habit and coated diamonds from the kimberlites of Zaire, Botswana [3], Yakutia [4], and Brazil [5]. In the diamond crystal described here, calcite forms peculiar rounded grains, mainly in the central part of the enstatite inclusion. These relationships can be probably interpreted as evidence for the initial entrapment of a silicate-carbonate melt, which separated into two phases during cooling.

It was experimentally shown that diamonds can crystallize directly from silicate-carbonate and carbonate melts, and it was supposed that such melts could serve as a diamond crystallization medium in the mantle [19]. It was also demonstrated that diamonds can be produced by the interaction of carbonates and silicates (in particular, enstatite) in the presence of C-O-H fluids [20].

Calcite microinclusions were rather widespread in three of the four samples. It should be noted that such inclusions have long been considered as epigenetic secondary phases in diamonds [1]. However, syngenetic calcite inclusions completely isolated in the host diamond were described in some transparent crystals [6, 18, 21]. It is reasonable to suggest that the carbonate phase was initially represented by aragonite, which crystallizes in the diamond stability field, and the aragonite was subsequently transformed into calcite.

The dark gray octahedral diamonds are similar to perfect crystals of octahedral habit in that they contain numerous sulfide inclusions. It is known that sulfides are the most abundant phases among macroinclusions in perfect crystals [1]. However, they are mainly represented by iron sulfides, whereas nickel sulfides were found in our samples. Among polycrystalline diamond aggregates, significant amounts of sulfide microinclusions were found only in fine-grained aggregates consisting of octahedral microcrystals from the kimberlites of China [11]. It is interesting that nickel sulfides were also predominant in these aggregates.

In contrast to macroinclusions in perfect diamond crystals, native metals, Fe-Cr intermetallic compounds, and metallic Fe are widespread among microinclusions in the dark gray crystals of octahedral habit. These phases were observed in all our samples. Inclusions of metallic iron were previously reported as macroinclusions in diamond crystals. However, among the tens of thousands of macroinclusions analyzed in diamonds, only a few grains of metallic iron have been found. Unusual spherical forms of metallic iron were described [22], and in some samples it was confined to the central (seed) parts of crystals [23].

In contrast, microinclusions of native metals were observed in most semitransparent and opaque polycrystalline diamond aggregates, most of which were gray or black in color and were previously studied by analytical electron microscopy. Inclusions of native Cr, Fe, Ni, and Cu were described in cryptocrystalline diamond aggregates from Yakutian deposits [10]. Cryptocrystalline diamond aggregates from Central Africa contained inclusions of metallic Fe, Cr, Ni, Ag, and Ni-Fe solid solution [8]. Native Au, Ag, Fe, Cr, Ti, Cu, and Au-Ag solid solution were detected in cryptocrystalline diamond aggregates from Brazil [9]. Inclusions of metallic Fe and Pb and Fe-Cr solid solution were found in fine-grained diamond aggregates from the kimberlites of China [11]. Metallic Fe, Cu, Ag, and Zn and Fe-Cr solid solution were detected among inclusions in coarse-grained diamond aggregates from Yakutian kimberlites [12]. Inclusions of metallic Fe and Cr and Fe-Cr and Fe-Cr-Ni solid solutions were found in diamonds of cubic habit from the kimberlites and placers of China [13, 14]. Metallic Fe and Cr and Fe-Cr and Cu-Zn solid solutions were investigated in rounded diamonds of dodecahedral habit from Australian lamproites [15]. Melts of transition metals (Ni, Fe, and Cr) are widely used in diamond synthesis [24]. They serve not only as carbon solvents but also exert a catalytic influence on the process of diamond formation. Their presence in natural crystals may be indicative of highly reduced conditions during diamond formation. On the other hand, inclusions of oxides (magnetite, ilmenite, etc.), carbonates, and other minerals are usually observed with native metals in distorted diamonds. The formation of such inclusion associations is probably related to nonequilibrium crystallization conditions and a change in the redox potential during crystallization. A possible explanation for the existence of such inclusion association is provided by the Taylor and Green [25] hypothesis on the formation of diamonds as a result of injection of deep reduced hydrocarbon fluids into deep-seated silicate rocks and their redox interaction. It should be noted that both reduced ( $\text{CH}_4$  and  $\text{H}_2$ ) and oxidized ( $\text{CO}_2$  and  $\text{H}_2\text{O}$ ) gases may be present in fluid inclusions from a single diamond [26, 27]. Tomilenko et al. [27] showed that the reduced fluids are confined to the central parts of crystals, whereas oxidized fluids were trapped by the outer zones. It is interesting that detailed studies of the distribution of mineral inclusions in perfect diamond crystals also indicated the confinement of reduced phases to the central parts of crystals [23].

Finally, it is noteworthy that many, if not most, researchers believe that natural diamonds were formed owing to the injection of fluids into deep-seated rocks. However, the characteristics of this complex dynamic process and the role of diamond crystallization in it are actively debated. Further studies are required to determine whether diamonds crystallized from hydrocarbon fluids [25] or from silicate-carbonate melts [2, 22] and brines [6], which could be formed during fluid interac-

tion with mantle rocks. It is also possible that all these processes were related to the formation of diamonds, which was responsible for the great diversity of both morphological types of diamonds themselves and inclusions in them.

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