===== PRODUCTION, STRUCTURE, PROPERTIES =====

Morphology of Diamond Crystals and Mechanism of Their Growth

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Abstract—Using the morphology of natural and synthetic diamond crystals as an example, the mechanisms of their growth of dislocation (spiral), non-dislocation (two-dimensional nucleation), normal (fibrous), and block (adhesive) character have been demonstrated. These mechanisms can be clearly seen in the morphological and microtopographic features of diamond polyhedra and xenocrystals. Growth occurs by the dislocation and normal mechanisms for most natural diamond crystals and the dislocation and two-dimensional nucleation mechanisms for synthetic diamond crystals.

INTRODUCTION

Diamond belongs to relatively rare minerals characterized by idiomorphism of precipitations. It most commonly forms just polyhedra and their intergrowths, polycrystals are less often, and xenomorphs are very rarely formed. Synthetic diamond also crystallizes in the form of polyhedra and their intergrowths. It is presumed that diamond idiomorphism is provided by strong covalent bonds in its structure. However, the faceting of its crystals is relatively deficient as compared to the faceting of ionic and even molecular crystals. As known, this is first of all governed by an intrinsic factor, namely, by the structure of a mineral or a synthetic crystal, i.e., by a high reticular density of planes and the rows of strong bonds within them. According to the P. Hartman theory on periodic chain bonds (PCBs), the simple forms and habit of a crystal are determined first of all by the set of continuous chains of strong bonds formed in its crystal lattice. Based on the analysis of PBCs in the structure of diamond, its crystals must be covered predominantly by octahedron flat faces in the process of dislocation or two-dimensional growth, i.e., crystals of octahedral habit must grow. However, the effect of external factors may cause the appearance of unconventional polyhedra and xenomorphic precipitations of diamond by the other mechanisms of growth. The morphological and microtopographic study of thousands of diamond microcrystals of diverse origin make it possible to illustrate their different growth mechanisms and show in what diamond they are inherent.

EXPERIMENTAL

The author had the possibility to study the morphology and microtopography of natural diamond crystals from kimberlites, lamproites, metamorphic rocks, impactites, placers, and macro- and microcrystals of synthetic HPHT and CVD diamond. Their morphological studies were carried out in the Semenenko Institute of Geochemistry, Mineralogy, and Ore Formation of the National Academy of Science of Ukraine on scanning electron microscopes (SEM) PEM-100 and JSM-6700F equipped with a JEOL JED-2300 energy-dispersive system for microanalysis (Japan). The SEM photos of diamonds were taken at an accelerating voltage of 15 kV (sometimes, 20 kV), a probe current of 6×10^{-10} A, and a probe diameter of 1—2 μm.

Table 1. Morphology of diamond crystals and mechanism of their growth

Diamond type	Simple crystal forms	Habit of crystals	Crystal growth mechanism
Mantle (kimberlite, lamproite, lamprophyre)	{111}, {100}, {110}, different {hhl}, {hkk}, {hkl}, and {hk0} on microcrystals	Octahedral, cubic, rhombic dodecahedral, combined {111} + {110}, {111} + {110} + {100}, and {111} + {100}	Dislocation (spiral), non-dislocation (two-dimensional nucleation), normal (fibrous), block (adhesive)
Mantle−metamorphic (metamorphic rocks)	{111}, {100}, {110}	Octahedral, cubic, combined {111} + {110}, {111} + {110} + {100}, and {111} + {100}	Dislocation (spiral), non-dislocation (two-dimensional nucleation), normal (fibrous), block (adhesive)
Mantle−ophiolite (peridotite-chromitite)	{111}, {100}, {110}, {311}	Cube-octahedral, octahedral	Dislocation (spiral), non-dislocation (two-dimensional nucleation)
Volcanogenic (eruption products of modern volcanoes)	{111}, {100}, {110}, {311}, {332}	Cube-octahedral, octahedral	Dislocation (spiral), non-dislocation (two-dimensional nucleation)
HPHT	{111}, {100}, {110}, {311}, {511}, {711}	Octahedral, cubic, combined {111} + {100}, {111} + {110} + {100}, {111} + {110} + {100} + {311}, trapezohedral	Dislocation (spiral), non-dislocation (two-dimensional nucleation)
CVD	{111}, {100}, {110}	Octahedral, cubic, combined {111} + {100}	Dislocation (spiral), non-dislocation (two-dimensional nucleation), adhesive
Impact, apographitic (impact rocks of meteorite craters)	{0001}, {10$\bar{1}$0}	Pinakoidal, prismatic-pinakoidal	Martensitic, recrystallization

RESULTS AND DISCUSSION

Table 1 contains the original and literature data on the simple forms and habits of natural and synthetic diamond crystals and the mechanisms of their growth. According to the geological and genetic type of diamondiferous parental and host rocks, natural diamond should be classified into diamond from mantle rocks, i.e., kimberlites, lamproites, and lamprophyres, diamond from metamorphic rocks, and diamond from impact rocks of Earth' meteorite craters. Diamonds from these rocks are the best studied ones. In addition to these rocks, some other ultrabasic and basic rocks are also lowly diamondiferous [1], but the diamonds of these rocks have not been sufficiently studied yet. In recent decades, there occurred a lot of papers on microdiamonds (of up to 0.5 mm in diameter) in such plutonian rocks as peridotites and chromitites from the Earth's ophiolite belts (from the Eurasian Alpine Belt or Alpine-Himalayan Belt, diamond occurrences from Myanmar, many places of Chinese Tibet, India, Turkey and Albania, and also from the Ural Caledonian Belt in Russia, and the Central Asian Caledonian Belt in China) and in the eruption products of modern volcanoes in Kamchatka from basalts, andesite basalts, tuffobreccias, and tuffs [2−4]. However, it should be immediately pointed out that the truthfulness of diamond finds in the ophiolite and contemporary volcanic rocks is put in doubt, and some researches consider them as synthetic HPHT diamond [5, 6]. Just the flat-faced morphology of their crystals and the inclusions of metals and alloys in them are pointed out as one of the important signs arguing for the contamination of samples from the aforesaid rocks with synthetic diamond crystals. Synthetic HPHT and CVD diamonds have their particular crystals morphology [7−9] contract to the morphology of natural diamond crystals. The crystals of these two synthetic diamonds different in synthesis conditions also appreciable differ from each other. Impact diamond has a radically different morphology, and crystals of this diamond are paramorphs with respect to the initial carbon material [10].

First of all, it should be pointed out that natural diamond crystals of mantle origin are characterized by that the growth of octahedron faces follows the so-called antiskeletal pattern, when the origination of new and new layers more often occurs in the central regions of faces instead of crystal apices and edges.
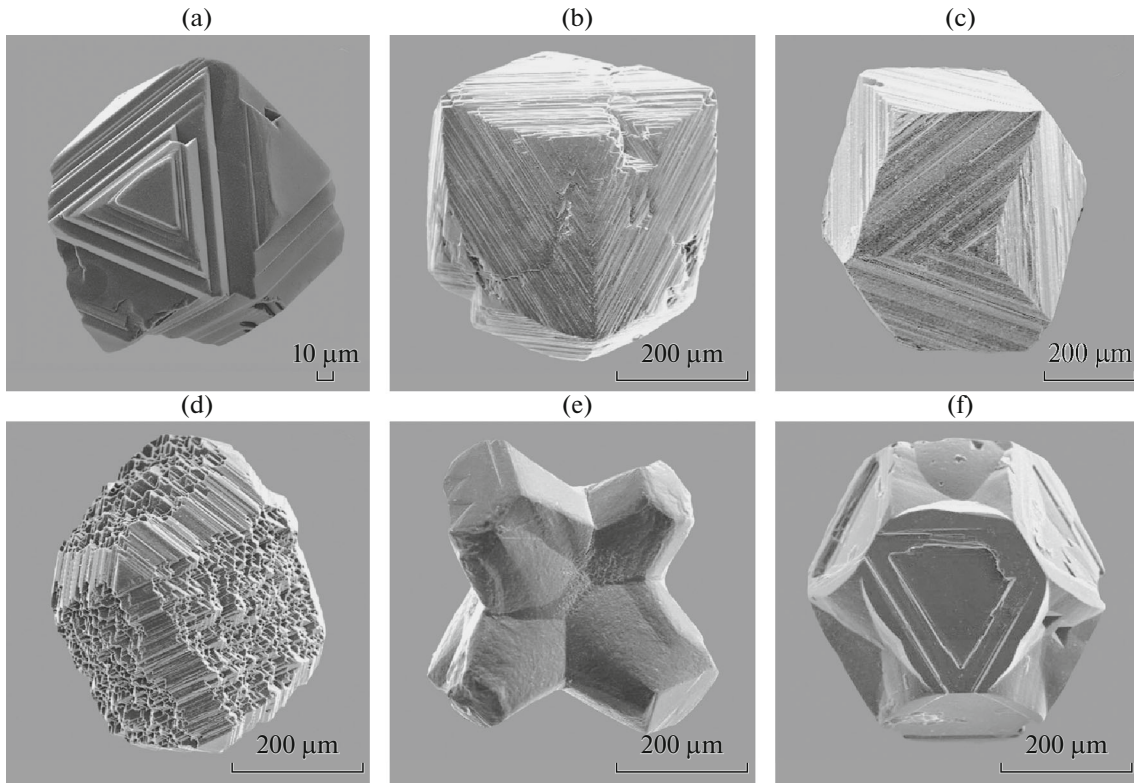
Fig. 1. Signs of antiskeletal and skeletal growth on diamond crystals from kimberlites and placers: (a) octahedron with gradual overgrowth along (111), (b) transitional octahedron-rhombic dodecahedron crystal form, (c) pseudo-rhombic dodecahedron, (d) pseudo-cube with miniature octahedron and pseudo-rhombic dodecahedron faces, (e) skeletal crystal appearing upon the degeneration of octahedron faces, (f) skeletal crystal with hollow octahedron and cube faces; (a) microdiamond from the Samotkan Neogene placer on the Ukrainian Shield, (b−f) microdiamonds from Yakutian kimberlites.

Most natural diamond crystals grow by this pattern to result in transitional diamond crystal forms {111} + {110} and pseudomorphs representing a kind of cubes in addition to some specific skeletal crystals (Fig. 1). This is also due to the fact that the rate of the overgrowth and outgrowth of octahedral faces on diamond crystals are close to each other or even the overgrowth rate is higher. Such a diamond crystal formation pattern was declared by Ansheles [11, 12], who called it antiskeletal growth. Typical skeletal polyhedra as such with hollow octahedron and cube faces are rarely encountered for natural diamond (Fig. 1f) as well as skeletal cubes with hollows faces for diamond macrocrystals from kimberlites. Such crystals grow when the growth layers appear at their edges and apices and do not completely grow into the center of faces. To exemplify the beginning of crystal growth from edges, it is possible to mention the formation of CVD diamond microcrystals, when poorly seen edges of a diamond octahedron first appear on a cluster of globules, being followed by the formation of its faces, and cube faces grow with a further decrease in the temperature (Fig. 2).

The antiskeletal pattern of the formation of most natural diamond crystals just evidences the dislocation mechanism of their growth. Additional evidences for such a growth mechanism are the microtopographic features of octahedron faces, i.e., the development of triangular straight parallel growth vicinals on them and their frequent polycentrism and, as a rule, numerous inversely parallel triangular pits on the same faces. These sculptures mark the area where dislocation develop and look as replacing the possible visible spirals of growth, whereas no sharply visible spirals of growth on natural diamond octahedron faces are known to the author. The author observed only a rare case of spiral growth for the cube-rhombic dodecahedron of diamond from kimberlites (Figs. 3a and 3b) with a spiral of growth on a cube face and numerous nano- and microblocks of growth on rhombic dodecahedron faces. Two mechanisms of growth—spiral and block—were surprisingly combined in the formation of this microcrystal.

Dislocation (spiral) growth is inherent in synthetic HPHT and CVD diamonds alongside with the non-dislocation (two-dimensional) mechanism of growth. This is especially typical for HPHT crystals. An evidence for the first mechanism of growth are frequent spirals on cube and octahedron faces in crystals of
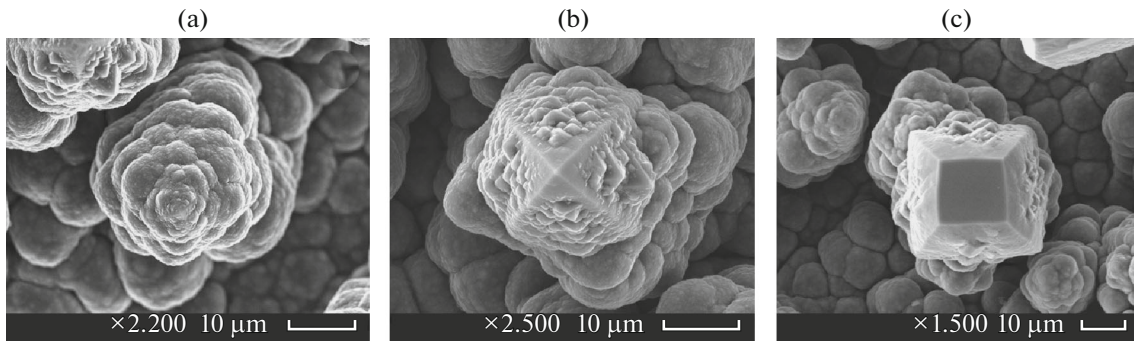
Fig. 2. Growth of CVD diamond polyhedra: (a) appearance of octahedron edges on clusters of globules, (b) formation of octahedron faces, (c) formation of a cube face.
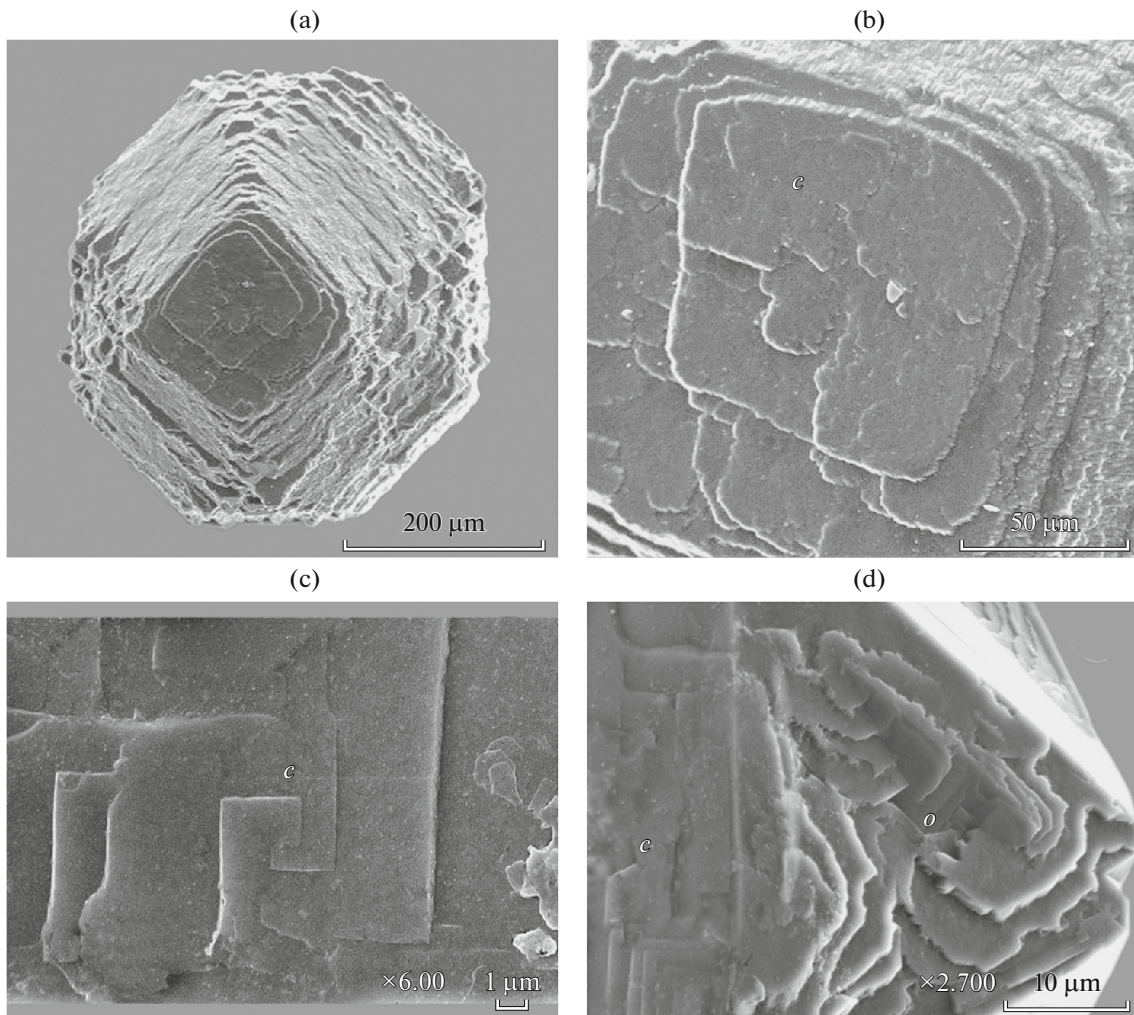

Fig. 3. Signs of spiral growth on diamond crystals of different origin: (a, b) spiral on a cube face of a microcrystal from Yakutian kimberlites, (c, d) spirals on a CVD microcrystal ((c) on cube face c, (d) on octahedron face o).

these synthetic diamonds, and the second mechanism are also indicated by frequent skeletal cube and octahedron faces on HPHT diamond crystals. The spirals of growth on cube and octahedron faces in a complex twin of CVD diamond crystals—a five-fold twin of cube-octahedra in the form of a "dipyramidal prismatic" crystal are shown in Figs. 3c and 3d. Each cube and octahedron faces are covered by several
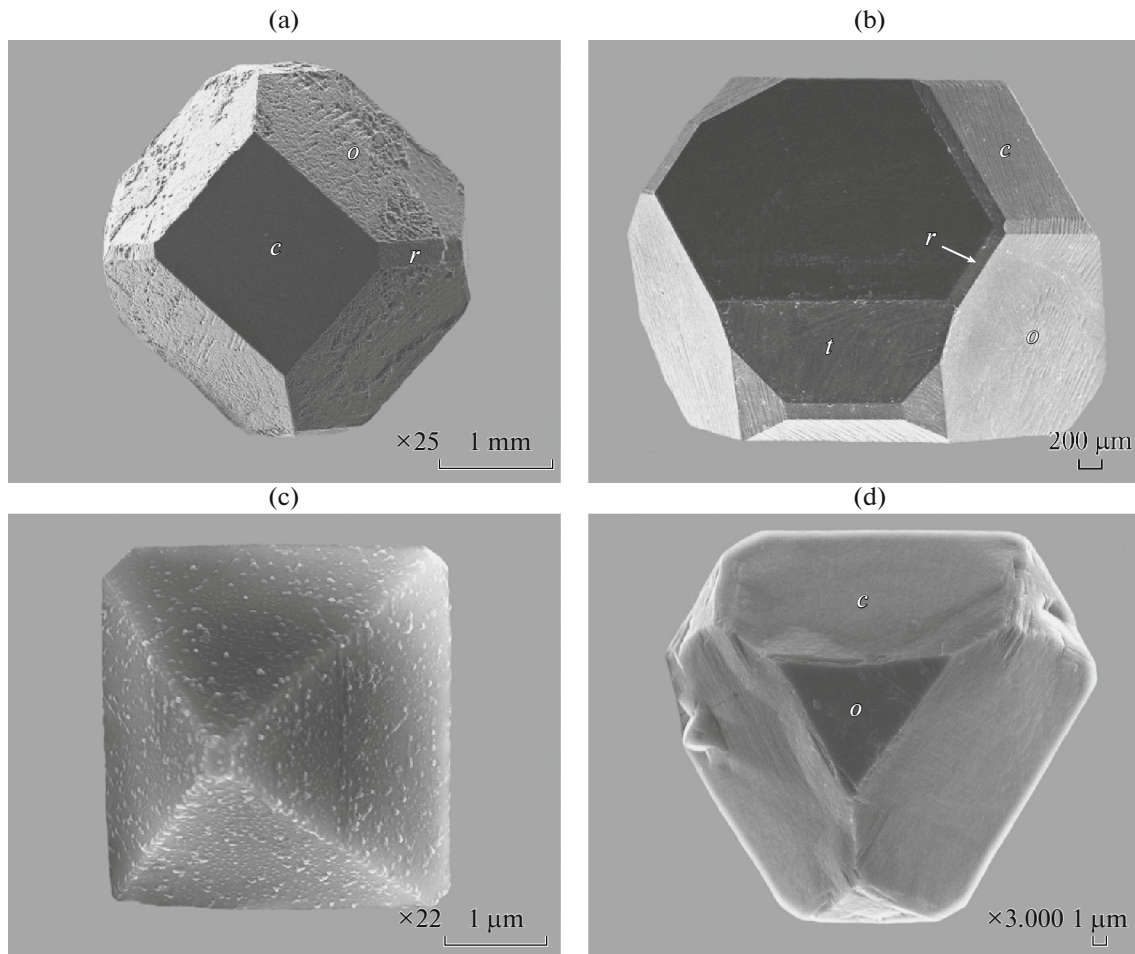
(a)

(b)

(c)

(d)

Fig. 4. Typical habits of crystals for HPHT (a). (b) and (c, d) CVD diamond crystals: *o* is octahedron, *c* is cube, *r* is rhombic dodecahedron, *t* is trapezohedron {311}. Diamonds were synthesized in the Bakul Institute for Superhard Materials of the National Academy of Sciences of Ukraine.

spirals. The height of these spirals is different, being larger on the octahedron faces. The symmetry of these spirals corresponds to the surface symmetry of cube and octahedron faces.

Microdiamond crystals from ophiolites and eruption products of modern Kamchatka volcanoes have the morphology inherent in synthetic HPHT diamonds [2−4] and slightly differ from the morphology of CVD diamonds by a high number of simple crystal forms (Fig. 4). They represent flat-faced crystals with cube and octahedron faces and, sometimes, with the development of narrow rhombic dodecahedron faces. Secondary crystal forms {311} and {332} were also goniometrically revealed on volcanogenic diamonds. The narrow faces of form {311} can also be seen on some crystals of ophiolite diamonds. However, no characteristic, but less widely encountered crystal forms of HPHT diamonds, such as trapezohedra {511} and {711}, have been detected on crystals of ophiolite and volcanogenic diamonds yet. Nevertheless, the morphological features of these diamonds allow us to believe that the mechanisms of their growth are the same as for HPHT diamonds: such crystals have grown by the dislocation (spiral) and non-dislocation (two-dimensional) mechanisms. The flat-faced cube-octahedral morphology of ophiolite and volcanogenic diamonds seems to be caused by the inclusions of different metals and alloys as catalysts for such growth of crystals. Let us also point out that the cube flat faces on kimberlite diamonds are rarely encountered, and such faces are secondary and only blunt the apices of fine octahedral crystals [13].

A major fraction of mantle diamond grows by the normal (fibrous) mechanism of growth. In contrast to kimberlite and lamproite diamonds, fine metamorphic diamonds are characterized by a great variety of crystallization forms, which have grown by the normal mechanism. Crystals of fibrous growth are predominantly cubes and cuboids (Fig. 5). Each fiber is laminated in the form of planes (111). Concentric zonality along (100) frequently exhibited in the sections of cubes is the crystallization front for a set of
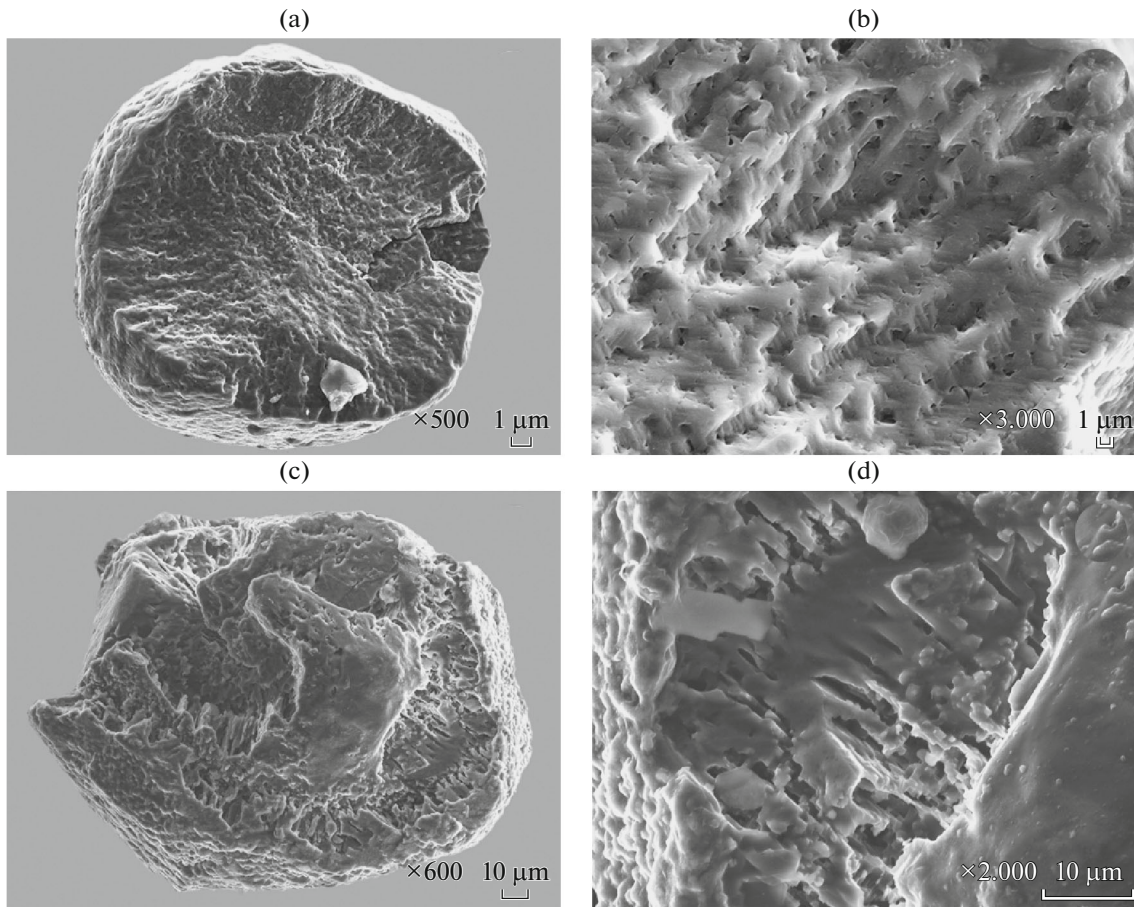
Fig. 5. Signs of normal (fibrous) growth for diamond crystals: (a, b) cuboid and its fibrous inner structure, (c, d) coat microdiamond with signs of two-stage growth in the form of a massive crystal structure under the coat and a fibrous structure of the coat; microdiamonds were taken from the Samotkan Neogene placer on the Ukrainian Shield.

fibers instead of the faceting growth of a crystal along (100). In other words, this zonality parallel to the cube sides is not faceting overgrowth in the direction of growth pyramid ⟨100⟩, but represents the crystallization front of a fibrous structure and sometimes has a wavy tortuous agate shape. The faces of such crystals always have a bumpy surface. It has been statistically shown on the basis of IR spectroscopy data that such crystals, as a rule, with fluid water inclusions are rich in hydrogen centers in contrast to the diamond crystals with anhydrous fluid inclusions, which have grown by the spiral and two-dimensional nucleation mechanisms. However, it has bon been clarified in what way these hydrogen centers promote the fibrous growth of cubic crystals. It is likely that they block the surface outgrowth of octahedron faces.

The block mechanism of growth is followed only by a small fraction of natural diamond crystals. This pattern of growth is most often typical for the cubes from kimberlites [14] (Fig. 6a). The very rare phenomenon of adhesion between globules with the formation of diamond octahedra and cube-octahedra was observed by the author in the Bilylivka meteorite crater on the Ukrainian Shield [15] when depositing nano- and microcrystals of globular diamond octahedra onto the surfaces of apographitic impact diamond (Fig. 6b). The adhesive mechanism of growth is followed by relatively rare xenomorphic diamond crystals (Figs. 6c and 6d).

The morphology of natural impact apographitic diamonds and the mechanism of their growth are quite other than for the polyhedra of mantle, metamorphic, ophiolite, volcanogenic, and synthetic diamonds (Fig. 7). These two- or three-phase crystals are often composed of lonsdaleite, residual or new graphite, and diamond itself. Such diamond results from the solid-phase transformation of the graphite structure into lonsdaleite and diamond by the martensitic mechanism. A lonsdaleite structure is formed upon the so-called longitudinal bending of graphite layers, and their corrugation gives a diamond structure [16]. The most powerful argument for the impact-metamorphogenic nature of such impact diamond is its apographitic morphology, surface microtopography of faces (0001) with new sculptures, a multi-
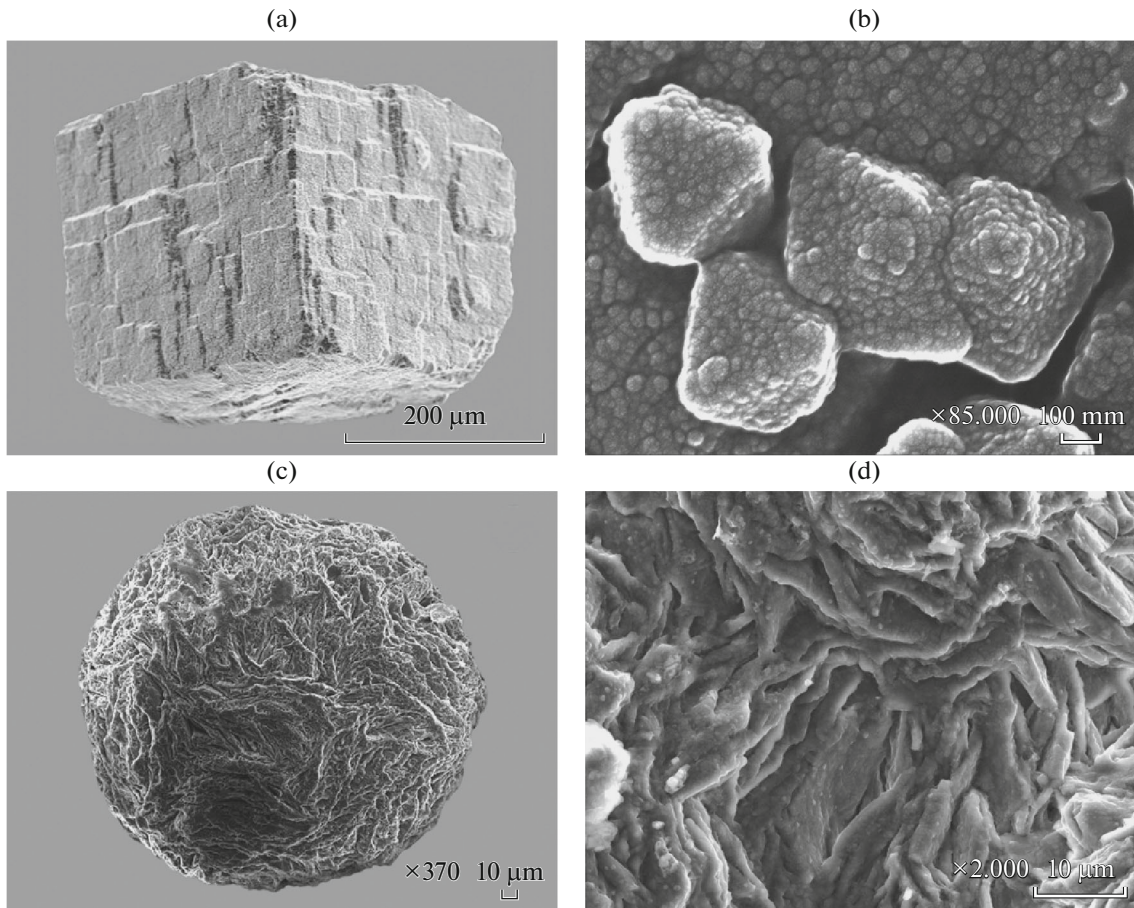
Fig. 6. Signs of block (adhesive) growth on diamond crystals of different origin: (a) growth of a cube by means of parallel rectangular microblocks, (b) growth of octahedra by means of globules, (c, d) growth of a roundish grain by means of distorted fibrillae; (a) microdiamond from Yakutian kimberlites, (b) nano-microdiamonds from the Bilylivka meteorite crater on the Ukrainian Shield, (c, d) microdiamond from the Samotkan Neogene placer on the Ukrainian Shield.

phase composition (diamond, lonsdaleite, graphite), regular structural relationships between these phases, an isotopic composition of carbon and its correspondence to isotopic composition of graphite carbon from the crater-forming rocks, the content and composition of impurities, specific physical properties, and theoretical calculations and experimental studies on the synthesis of such diamond. Impact apocoal and apowood substance diamonds called togorite and karite, respectively, are also known [17, 18].

The cited examples of mechanisms for the growth of diamond polyhedra and xenocrystals almost completely correspond to the morphological concept, which is proposed by Sunagawa [19, 20] for the specific features of the growth of synthetic and natural diamonds and can be called genetic (Fig. 8). It is based on the theoretical analysis of the diamond crystal growth rate depending on the ratio between the driving forces of crystallization (oversaturation, overcooling, and growth surface character (rough or smooth), and growth mechanism type (dislocation spiral, non-dislocation two-dimensional, and adhesive)). This development of I. Sunagawa can be applied to any types of natural and synthetic diamond. He has distinguished two threshold levels for the driving forces of crystallization, such as $\Delta\mu/KT^*$ and $\Delta\mu/KT^{**}$, at which the growth surface character, the growth mechanism type and, correspondingly, the morphology of crystals prove to be different. It is implied that the growth surface will be rough to implement the adhesive growth mechanism resulting in dendrite crystal forms and polycrystals and, with a further increase in the crystallization driving force, in spherulites, if this level is higher than $\Delta\mu/KT^{**}$. The growth surface will be smooth below the level of $\Delta\mu/KT^{**}$, and the two-dimensional nucleation mechanism is implemented between $\Delta\mu/KT^*$ and $\Delta\mu/KT^{**}$ predominantly along the edges of crystals and, as a result, skeletal crystals or crystals with a funnel-like structure of faces may be formed. Below $\Delta\mu/KT^*$, the only crystal growth mechanism is the dislocation spiral mechanism, when growth occurs on a smooth surface, and growth layers propagate from the center to periphery of faces. This is the way, in which polyhedra with
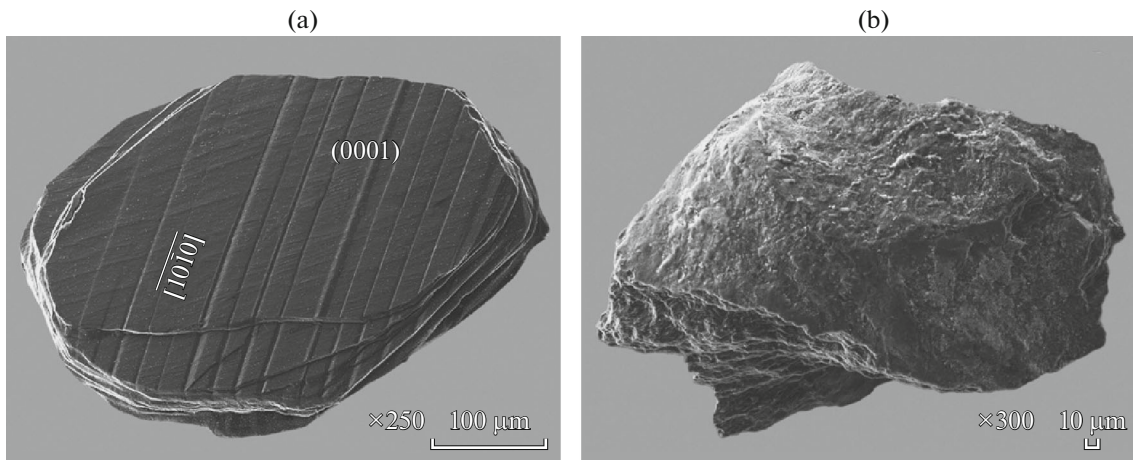
(a)   (b)

Fig. 7. Impact diamonds: (a) apographitic diamond from the Popigai meteorite crater (Siberia), (b) apographitic diamond from the Tunguska disaster site (Siberia).
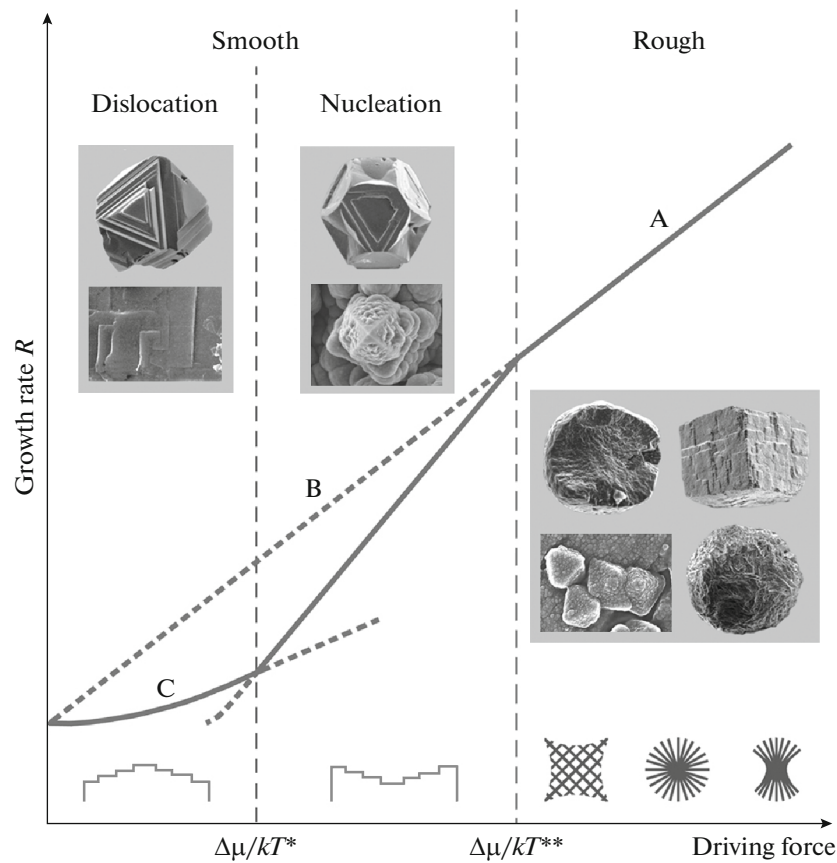

Fig. 8. Diagram proposed by Sunagawa with our marks for diamond crystals of different growth mechanism and origin.

simple symbols of faces are formed. The variations of crystal habits are determined by the relative normal growth rates on certain smooth surfaces.

Based on such theoretical analysis, Sunagawa has classified the morphology of natural diamond into three major categories. The first category incorporates single crystals (octahedra, transitional crystal forms like an octahedron-rhombic dodecahedron, and their intergrowths) formed as a result of spiral

growth or by the two-dimensional nucleation mechanism under the action of a driving force weaker than for $\Delta\mu/KT^{**}$ and usually weaker than for $\Delta\mu/KT^*$. The second category includes polycrystalline aggregates (bort, ballas, and carbonado) formed under the conditions of driving forces above $\Delta\mu/KT^{**}$. These diamonds are formed by adhesive growth mechanism on a rough surface. The third category is formed by crystallization diamonds (coat diamonds, cuboids), which have sustained the change of crystallization conditions (driving forces): they grew first as single crystals under the conditions below $\Delta\mu/KT^*$ and, probably, even below $\Delta\mu/KT^{**}$ and further as polycrystals under the conditions above $\Delta\mu/KT^{**}$. Sunagawa has also analyzed the topographic character of diamond growth surfaces, in particular, their roughness depending on the conditions of growth. In the silicate medium of the growth of mantle diamond, only octahedron faces behave as smooth surfaces, on which spiral growth occurs. Cube faces appear only under the conditions of crystallization driving forces above $\Delta\mu/KT^{**}$ and behave as a rough surface in the silicate medium of magma. Faces {100} have no spiral growth layers, and their surface is rough or bumpy. In contrast, not only an octahedron face, but also a cube face on synthetic diamond crystals synthesized in a metal−carbon system may behave as a smooth surface due to the surface reconstruction of faces {100}: rough cube face K is transformed into smooth cube face F. In the opinion of Sunagawa, this surface reconstruction occurs in such a synthetic solution-melt, whose metallic element as a solvent has a small ionic radius, whereas this does not occur in a natural silicate solvent with a large ionic radius. For this reason, synthetic diamond crystals demonstrate spiral growth on both octahedron and cube faces. Synthetic HPHT diamonds grown in a silicate, carbonate, or other medium without metal solvents get an octahedral form similar to natural diamond crystals [21]. At the same time, the application of different metal solvents leads to that the habit of crystals is changed from a characteristic cube-octahedron even to a rhombic dodecahedron and a trapezohedron. However, the reason for the appearance of synthetic crystals with a trapezohedral, rhombic dodecahedral, or combined habit {111} + {100} + {110} + {311} has not been clarified. But microdiamonds exhibit some deviations from the concept of Sunagawa, which can be clearly seen on mantle diamond crystals. Thus, for example, the fact that flat faces of cubes and other simple crystal forms are observed on kimberlite microdiamonds may be classified as a contradiction [13] or, for instance, the spiral growth of cube faces on a mantle diamond microcrystal (see Figs. 3a and 3b). On the whole, it is possible to point out that there is the following trend in the development of diamond crystallization forms from mantle or metamorphic rocks: when the depth of crystallization increases, the shape of diamond crystals is simplified to the most stable octahedral form of growth. The same trend is demonstrated by a decrease in the content of impurity nitrogen and hydrogen centers and an increase in the C^{12} isotope content in diamond crystals.

CONCLUSIONS

The analysis of data on the mineralogical crystallography of diamond indicates that the two main patterns of its crystallization are implemented in the nature, such as free growth representing diffusion crystallization and, in the case of impact diamond, forced growth under impact loading, i.e., the solid-phase transformation of graphite into diamond, and several crystal formation mechanisms, such as dislocation (spiral), non-dislocation (two-dimensional nucleation), normal (fibrous), and block (adhesive) mechanisms for mantle diamond and recrystallization mechanism for impact diamond.

The hypothesis of mantle diamond crystallization from a fluid in a relatively free space (predominantly, in a macroscopic cavities and cracks) may explain idiomorphism for the overwhelming majority of its crystal forms.

The reason for the predominant antiskeletal formation of diamond octahedra is not quite clear: why a crystal face is most often formed from its center instead of edge. This may be due just to the predominant dislocation mechanism of the growth of diamond crystals.

It is remarkable that the faceting of mantle diamond is simplified with an increase in the size of crystals: a crystal is usually covered with octahedron faces. Such a "morphologically dimensional" phenomenon is typical for crystals of many minerals and synthetic crystals of many substances: crystals grow due to the enlargement of structurally significant faces.

Hence, it can be stated that

—Mantle diamond crystals from kimberlites, lamproites, and lamprophyres grow predominantly by the dislocation mechanism, more rarely by the normal mechanism, and rarely by the non-dislocation two-dimensional nucleation mechanism;

—Metamorphic diamond crystals grow predominantly by the normal mechanism, more rarely by the dislocation mechanism, and rarely by the non-dislocation two-dimensional nucleation mechanism;

—Synthetic diamond crystals grow predominantly by the dislocation and non-dislocation two-dimensional nucleation mechanisms; and

—Impact diamond crystals appear during the recrystallization of carbon substance due to its structural reorganization under the action of high pressure and temperature.

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