MINERALS
AND MINERAL PARAGENESES

Comparison of Diamonds from the Rassolninskaya Depression and Modern Alluvial Placers of the Krasnovishersky District (Ural Region)

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Abstract—Three hundred thirty-six diamonds from deposits of the Rassolninskaya depression and 144 crystals from recent alluvial placers of the Krasnovishersky district were studied by IR absorption and photoluminescence spectroscopy. It is shown that crystals from the Rassolninskaya depression have a close-to-normal distribution for the nitrogen concentration. The average nitrogen content is 725 ppm, and no nitrogen-free crystals were detected. A sampling from recent alluvial placers contains 25% crystals with a nitrogen concentration smaller than 150 ppm; 3% of them are nitrogen-free. Among crystals from the Rassolninskaya depression, 12% are octahedral, 80% rhombododecahedral, and only one crystal has relics of cubic faces. The collection from recent placers contains 3% cubic crystals, 10% individuals with relics of cubic faces, 16% octahedroids, and 66% dodecahedra. Alluvial diamonds are often encountered with crescent-shaped cracks; however, they were observed only on a single crystal from the Rassolninskaya depression. It has been revealed that among alluvial placer diamonds, up to 95% of crystals contain nitrogen in the form of B1 defects. Thus, first, in morphological and structural-mineralogical features, diamonds from the Rassolninskaya depression differ from crystals of the nearest recent alluvial placers; second, they may belong to primary deposits based on the set of their characteristics.

INTRODUCTION

Structural and mineralogical studies of diamonds are the most important procedure at diamondiferous sites with debatable sources. One of these sites is the Rassolninskaya depression (hereinafter, RD) in the Krasnovishersky District, whose intrusive pyroclastites, which in composition correspond to low-alumina lamproite (Lukyanova et al., 2000), are considered by many researchers as the diamond-bearing source rocks (Anfilogov et al., 2007; Lukyanova et al., 2011). A characteristic morphological feature of RD diamonds and Ural crystals as a whole is the sharp dominance of dodecahedroids (Kukharenko, 1955). Many researchers have explained this type of crystals as the dissolution of table-faced individuals (Fersman, 1954; Kukharenko, 1955; Orlov, 1973; Moore and Lang, 1974). Anatomical studies in recent decades (Beskrovanov, 1992; Davies, 1999; Thomson et al., 2014) and high-temperature diamond dissolution experiments (Khokhryakov, 2000) have proved that most rounded crystals were subjected to dissolution. However, the anatomical peculiarities of some crystals visualized by cathodoluminescence (Harte et al.,

1999; Wright, 2003; Gaillou et al., 2012) indicate the possible growth nature of rounded forms at certain crystal formation stages, which very rarely appear in their morphology .

For a long time, crystals from Ural placers could only be studied morphologically (Kukharenko, 1955; Orlov, 1973; Shemanina, 1993; Shafranovsky, 2001; Posukhova, 2007; Rakin 2013). G.I. Shafranovsky (2001), N.N. Zinchuk, and V.I. Koptil (2003) previously gave a morphological description of crystals from the RD collection of JSC Geokarta-Perm. According to Koptil, among RD crystals (361 crystals were studied), 70% are cryptolayered dodecahedroids, 5% are octahedra and octahedroids, 18% are rounded with shagreen and plastic deformation bands, 2% are hemimorphic, and 0.3 are cuboids. Koptil considers the lozengelike sculpture of the studied crystals typical and notes isolated cases of crescent-shaped cracks and "abrasion" wear. In alluvial placer diamonds from the JSC Geokarta-Perm collection, Koptil identified the same occurrence frequencies of morphological types, but noted an increased (up to 20–40%) proportion of crystals with abrasion and pitting on edges. According

to Shafranovsky (2001), among RD crystals and alluvial placers, 80% are dodecahedroids, 6% octahedra, and 7% dodecahedroids with relicts of cubic or octahedral faces. Shafranovsky noted no traces of alluvial transport on RD crystals. V.I. Rakin conducted a detailed goniometric study of RD crystals and published his results in a monograph (Rakin, 2013). It is noteworthy that Rakin revealed signs of mechanical wear in 88% of RD samples.

Structural—mineralogical studies are singular and fragmentary, in particular, luminescence (Gomon, 1966; Isaenko, 2016) and infrared (IR) absorption studies (Kaminsky, 2001; Khachatryan et al., 2004; Lyutoev et al., 2012; Vasiliev et al., 2013; Fedorova et al., 2013). The most interesting comprehensive study (Laiginhas, 2008) includes infrared spectroscopy of diamonds from the Uralalmaz industrial mining company and an analysis of their inclusions. In (Laiginhas, 2008), a diamond age of 1.3 Ga was determined from the Re—Os ratio in sulfide inclusions, and a diamond formation temperature of 1050—1300°C was determined from mineral geothermometers; this range was also obtained from the equilibrium isotherms of nitrogen defect transformations.

Based on IR spectroscopy of the set of nitrogen defects, diamonds are divided into the following types: IIa, nitrogen-free; Ib, with low-temperature C defects; 1aA, with A defects; type 1aAB, with A and B1 defects; Type 1aB, with high-temperature B1 defects (Zaitsev, 2001). Type 1aAB crystals from various deposits have been compared by analyzing the distribution of B1 defects and the total nitrogen concentration (Taylor et al., 1990; Mendelsohn et al., 1994), the absorption coefficient of the 3107 cm^{-1} band (Khachatryan, 2009), and the absorption coefficient and position of the B2 band maximum (Bogush et al., 2009).

In diamond studies, photoluminescence (PL) spectroscopy is widely used, which can reveal a large number of various systems (Zaitsev, 2001; Yelisseyev et al.; Dishler, 2012). The advantage of luminescence spectroscopy is the large number of luminescence systems and high sensitivity in identifying impurity and intrinsic defects in the crystal structure; drawbacks are the qualitative character of the study, strong defect interaction, and nonuniform distribution of luminescence centers over the bulk of crystals.

The paper presents brief results from a study and comparison of 336 diamond crystals from the RD recovered in 1995–1999 (Geolkarta-Perm LLC collection) and 144 crystals from modern alluvial placers (APs) of the Bolshoi Kolchim and Bolshoi Shchugor rivers of the Krasnovishersky district (VSEGEI collection). We have also studied crystals of modern alluvial deposits of the Vizhay (14) and Usva (15) rivers in the Gornozavodsky district (VSEGEI collection). Our study and comparison of the spectroscopic characteristics of diamond crystals from the RD and APs of the Bolshoi Kolchim and Bolshoi Shchugor river basin represent the first time such investigations were carried out.

IR absorption spectra were recorded on a Vertex 70 spectrometer with a Hyperion1000 microscope with a resolution of 2 cm^{-1} and averaging of 32—200 scans in the range of 600—7000 cm^{-1}. The optical density spectra were normalized based on intrinsic two-phonon absorption; the nitrogen concentration in the form of A (N_A) and B1 (N_{B1}) defects and total concentration (N_{tot}) were calculated using the known proportionality coefficients (Boyd et al., 1994, 1995). In addition to the concentration of A and B1 defects, the absorption coefficient of the B2 band (a_{B2}) and the position of its maximum (v_{B2}) were determined, as well as the absorption coefficient of the 3107 cm^{-1} band (a_{3107}), corresponding to the V_3NH defect (Goss et al., 2014). The PL spectra were recorded on a Renishaw In Via spectrometer at 77 K with 488 and 785 nm laser excitation and a x5 objective in the 490—1050 nm range, as well as on a Horiba FL3 spectrometer at room temperature with excitation by a 450 W xenon lamp.

MORPHOLOGICAL FEATURES

In the brief description of crystals, we sought morphological features that correlate with structural—mineralogical characteristics. Since the dodecahedroid—tetrahexahedroid combination (Fig. 1h) is the limiting dissolution shape (Khokhryakov et al., 2002), it is often impossible to determine the shape of the original crystal from morphological features alone. If relicts of octahedral faces with characteristic trigonal etching pits are preserved or layers along {111} (Fig. 1h) are obvious on the dissolution surface, then it can be stated that the original crystal shape was an octahedron. If relicts of cubic faces with characteristic tetragonal pits (Fig. 1b) are preserved, then the initial form of the dissolved individual can be considered a cube. However, these signs are often absent on dodecahedroids, in which case growth forms of a crystal can be established only by studying its anatomy or spectroscopic characteristics. Therefore, we isolated crystals with octahedral and cubic faces (Fig. 1a), dodecahedrons with relicts of cubic faces (Fig. 1b) or octahedrons and polycentric, combinatorial, or transitional forms. Some cubic crystals had a yellow color due to the low concentration of C defects, two crystals had a yellow color due to N3 defects, and some were colorless, of type IaAB1 (Figs. 1e—1i). Figures 1a—1d demonstrate the evolution of the dissolution shape of a cubic crystal in the series *cuboid → dodecahedroid with relicts of cubic faces → dodecahedroid*. Two rounded dark green crystals were found in the AP collection, on the cleavage surfaces of which a coat is visible. In the IR range, these crystals are opaque and, in morphology, correspond to Yu.L. Orlov's four varieties. Additionally, obvious signs of wear were noted in
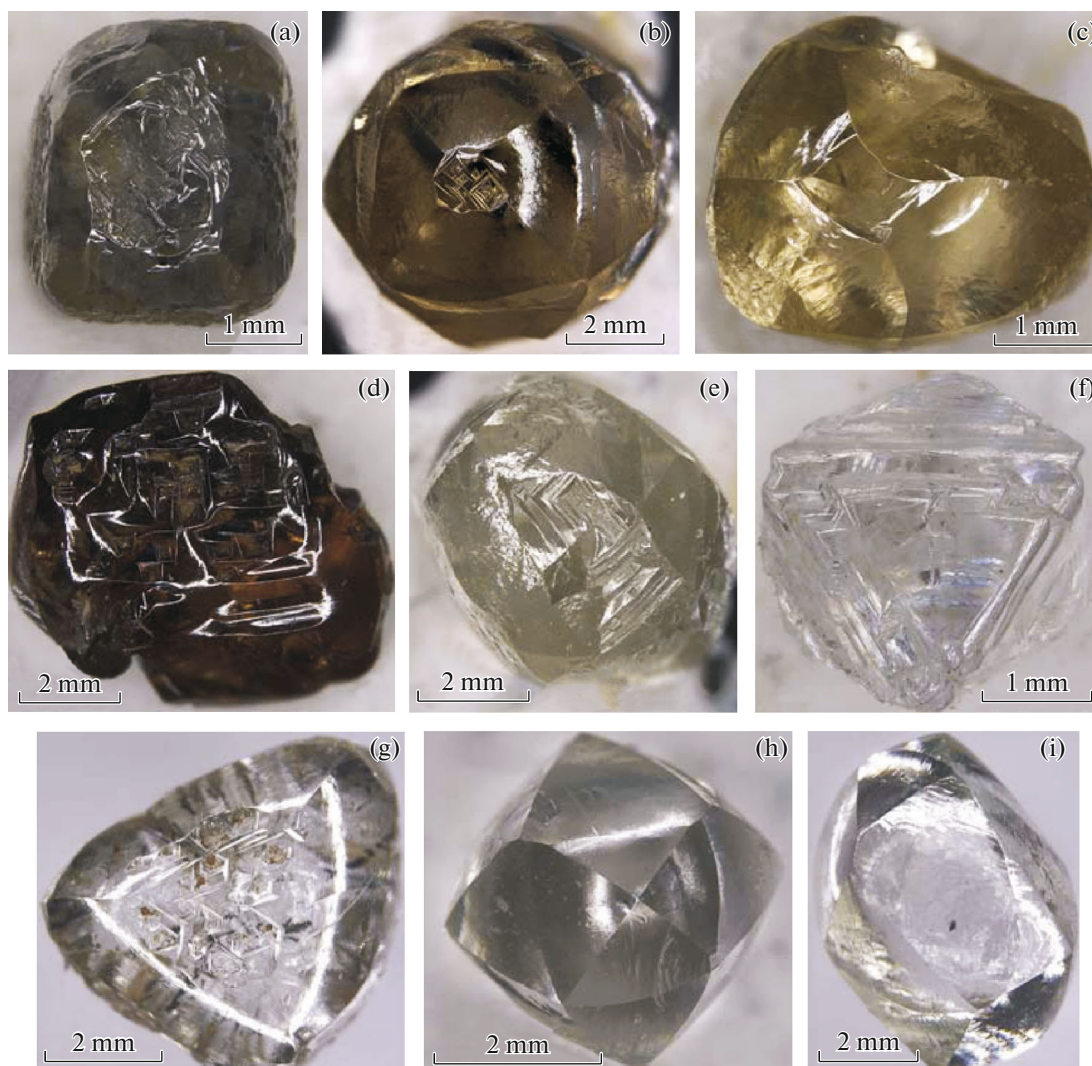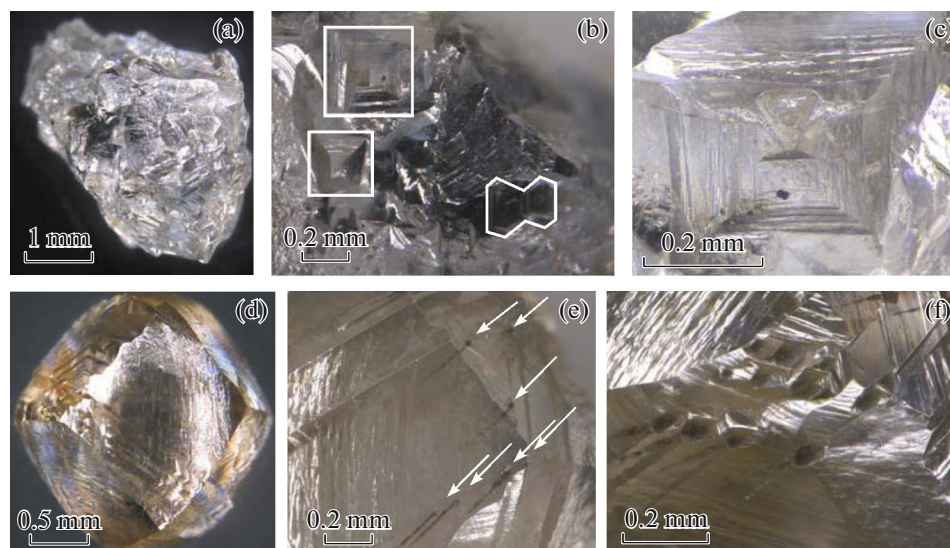
Fig. 1. Photographs of crystals from alluvial placers (a−f) and Rassolninskaya depression (g−i).(a) crystal with 601-66 cubic habit, type IaA, with surfaces of tetrahexahedroid and tetragonal pits, poorly pronounced lozengelike sculpture; (b) dodecahedroid tetrahexaedroid 123-76 with relicts of cubic faces, type IaAB, color caused by N3 defects; (c) dodecahedroid 158-76, type IaA, with slightly pronounced lozengelike sculpture; (d) crystal 223-76 with relicts of cubic faces, type Ib-IaA, with developed lozengelike sculpture; (e) dodecahedroid 122-76 with relicts of cubic faces, type IaAB; (f) laminar octahedron 16-552; (g) octahedroid, flattened along the L3 axis; (h) dodecahedroid tetrahexaedroid, result of dissolution of isometric octahedron; (i) typical Ural-type dodecahedroid.

the form of hatched faces, crescent-shaped cracks, and lozengelike sculpture. Figures 1g−1i show photos of some typical RD crystals, which are attributed to the Ural type, which dominate both the RD and AP collections

Nitrogen-free diamonds are a separate morphological type, characterized by polycentric growth (Fig. 2a). As they dissolve, tetragonal, multidirectional trigonal, and hexagonal etching pits develop on the surface (Figs. 2b, 2c). Earlier it was shown that during dissolution, on {111} faces, inversely oriented trigonal pits form (Khokhryakov et al., 2002). Apparently, this is true only for octahedral crystals; in nitrogen-free individuals with polycentric growth, various dissolution

sculptures are possible, including multidirectional trigonal, as well as their combination: hexagonal pits. Acicular etching channels along the intersection lines of twinning planes (Rose channels) have been noted on a single AP crystal with very pronounced deformation hatching. The etching channels have depths of 0.1−0.9 mm and are marked by hexagonal pits where they reach the surface (Figs. 2d−2f).

The results of the brief morphological description are summarized in Table 1. Dodecahedroids dominated the RD crystal sampling, 5% octahedra were noted, and one crystal with relicts of cubic faces was found, which matches Koptil and Shafranovsky's observations. The AP sampling is sharply distin-

Fig. 2. Photographs of crystals from alluvial placers. (a) General view of nitrogen-free crystal 614-66; (b) surface fragments with tetragonal, hexagonal, multidirectional trigonal pits (outlines circled); (c) a fragment of surface with trigonal figure on wall of tetragonal pit; (d) general view of crystal 16-658 with developed deformation hatching; (e) etching channels along lines of intersection of twinning planes (shown by arrows); (f) hexagonal cavities at exit of etching channels.

guished by a high proportion of cuboids and crystals with relics of cubic faces, 12% in total. One-fifth of the AP crystals have crescent-shaped cracks, which contrasts sharply with single RD crystals with this feature. Samples from the Gornozavodsky district are similar in morphological features to AP crystals.

IR SPECTROSCOPY RESULTS

Figure 3 shows the distribution of the nitrogen concentration and share of nitrogen in the form of B1 defects for the RD and AP crystals. In the RD sampling, there are no nitrogen-free type IIa crystals in the physical classification. As well, in the sampling from this source, no crystals were detected with extremely low- or high-nitrogen contents in the form of B1 defects—types IaA and IaB. The majority of crystals have a 30−60% degree of aggregation and 400−900 ppm total nitrogen concentration. The AP sampling differs starkly in that the crystals occupy the entire range of the diagram and are distributed fairly uniformly. Of the AP crystals, only half are in the same region of the diagram where most of the RD crystals are located. The AP sampling contains four nitrogen-free type IIa crystals (3%), 20 type IaA samples without B1 and B2 defects (14%), and four type IaB crystals (3%). Based on the model temperature, these crystals fall in the range of 1000−1400°C (Fig. 3).

The RD crystals have a close-to-normal unimodal distribution of the nitrogen concentration (Fig. 4a). In the AP sampling, one-fourth the crystals have N_{tot} less than 150 ppm and about 45% of crystals have a reduced nitrogen content compared to the RD sampling. The distribution of a_{3107} has a lognormal character, and the RD crystals are shifted toward larger values (Fig. 4b). For RD crystals, the arithmetical average of a_{3107} is 2.8 cm^{-1}, and the mode is 1.1 cm^{-1}; for AP crystals, these values are 1.1 and 0.44 cm^{-1}, respectively.

The smaller value of the a_{3107} average in the AP sampling compared to the RD sampling corresponds to a lower nitrogen concentration. The maximum a_{3107}

Table 1. Some morphological peculiarities of diamond crystals from Rassolninskaya depression and recent alluvial placers

Source	Octahedra, octahedroids, %	Dodecahedroids, %			Cuboids, %	crescent-shaped cracks, %	PDB, %
		relics of octahedral faces	without relics	relics of cubic faces			
Rassolninskaya depression	9	4	80	0.3	−	1	7
Alluvial placers	16	9	57	10	2	20	20

*PDB, plastic deformation band in absorption spectra.
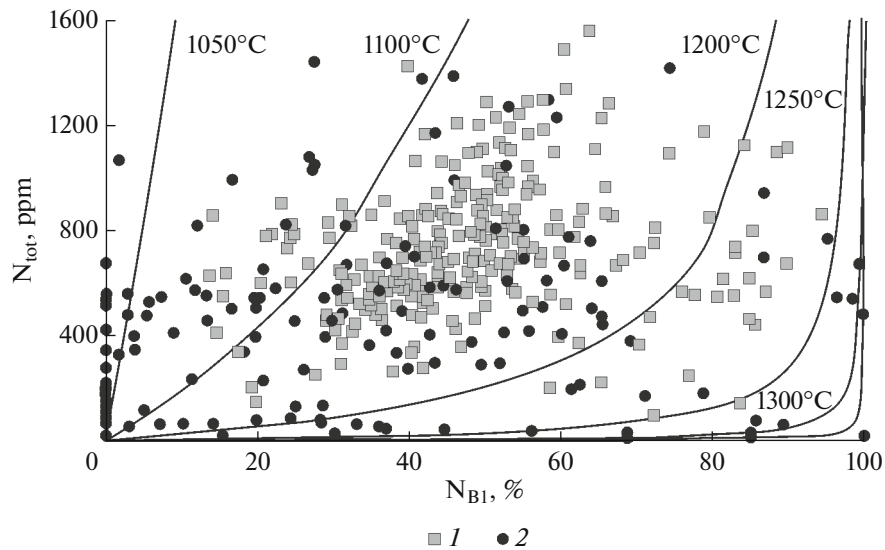
Fig. 3. Distribution of crystals of Rassolninskaya depression (*1*) and alluvial placers (*2*) in terms of nitrogen concentration N_{tot} and proportion of nitrogen in form of B1, N_{B1} defects. Isotherms corresponding to annealing time of 3 Ga are plotted on diagram (Taylor et al., 1990).
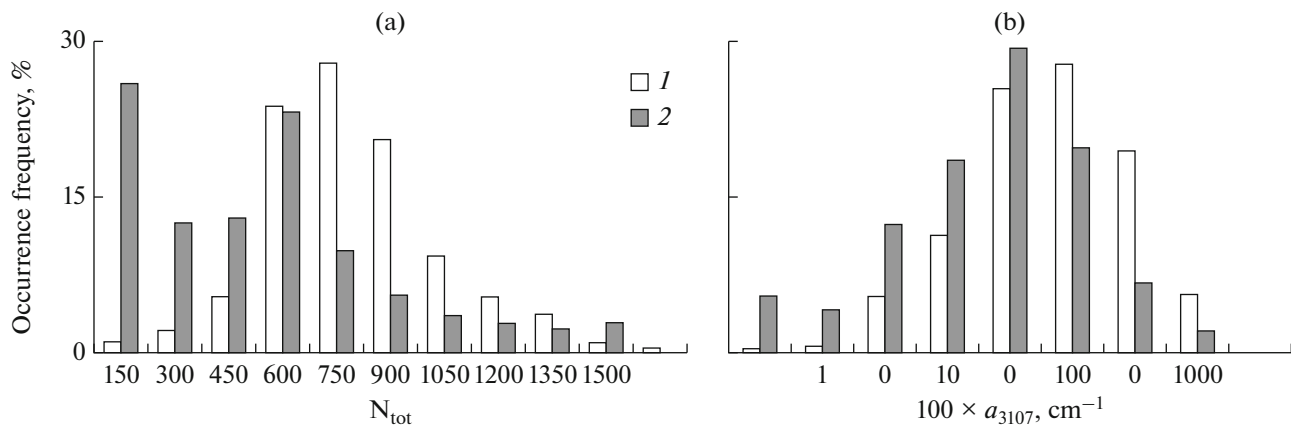
Fig. 4. Distribution of crystals of Rassolninskaya depression (*1*) and alluvial placers (*2*). (a) Nitrogen concentration N_{tot}; (b) absorption coefficient of hydrogen-related defect band a_{3107} in log–log coordinates.

values in the RD crystals were 17, 24, 25, 29 cm^{-1}, and in the AP sampling, 10 and 14 cm^{-1}.

Most of the AP crystals with an octahedral habit are characterized by a nitrogen content less than 600 ppm, and the B1 nitrogen distribution is uniform. AP dodecahedroids with relicts of octahedral faces are close to octahedral crystals in the analyzed parameters. There are type IaA crystals among the dodecahedroids with relicts of cubic faces, but there are also individuals with >40% nitrogen in the form of B1 defects and one crystal with >95% N_{B1}. In the entire AP sampling, 12% of cuboids and crystals with relicts of cubic faces are noted, half of which are type IaAB.

Some of these crystals are yellow due to absorption of the N3 system. In the RD sampling, there were no significant differences in nitrogen concentration and degree of aggregation between dodecahedroids, octahedroids, and octahedra.

Among the 14 Vizhay River crystals, five (35%) are attributed to physical type IaA, two are close to type IaB1, and one is low-nitrogen. For three samples, a_{B2} is more than 27 cm^{-1}, and for two samples, a_{3107} is more than 22 cm^{-1}. Among the 15 crystals of the Usva River sampling, three crystals (20%) are attributed to type IaA, and one, to type IaB1. Crystals of these types were not found among the 336 RD samples.

Table 2. Set of systems in PL spectra of diamond crystals from Rassolninskaya depression and from recent alluvium placers (excitation 488 nm)

Source/Luminescence band	H4	H3	GR1	575 nm	787 nm	926 nm	933 nm	max 720*	max 900*
Occurrence frequency, %									
Rassolninskaya depression	11	63	33	5	64	22	66	23	45
Alluvial placers	11	86	25	12	23	13	15	23	8

*max 720, max 900—wide luminescence bands with maxima at 720 and 900 nm.

LUMINESCENCE SPECTROSCOPY RESULTS

The PL spectra of 85 RD crystals and 140 AP crystals were recorded at room temperature with 360 nm excitation (Xe lamp). Under these conditions, the N3 system appears in and dominates the PL spectra of all RD crystals, and the H3 or H4 systems are observed in the spectra of 75% of samples. A wide structureless luminescence band with a maximum around 545 nm caused by S1, S2, and S3 centers was recorded in the spectra of 10% of crystals—the same proportion of crystals in whose spectra a system with a phonon-free line of about 582 and phonon lines at 605 and 626 nm is observed. In the AP sampling, the N3 system was recorded in the spectra of 95% of crystals, and 80% of the samples dominate the spectra. The broad PL band of the S1, S2, and S3 centers is observed in the spectra of 16% of AP crystals; 10% of the crystals dominate the spectra. About 40% of the AP crystals in whose PL spectra photoluminescence of the S1, S2, and S3 systems is recorded are cuboids or dodecahedroids with relicts of cubic faces; the rest are dodecahedroids. The N3 system is recorded in the PL spectra of 85% of cuboids and dodecahedroids with relicts of cubic faces. The set of luminescence systems for excitation by a 488 nm laser and the boiling temperature of liquid nitrogen is much more diverse. Under these conditions, broad luminescence bands are observed with maxima at 720 and a set of phonon replicas at 610, 618.5, 627, 636, 645.5, 655.5 nm (phonon energy of 0.029 eV), and a maximum of about 900 nm and phonon replicas at 729, 754, 779, 810, 838, 873 nm (phonon energy of 0.055 eV). Table 2 shows the occurrence frequency of the most intense luminescence systems: H3, H4, GR1, 575 nm and the 700.3, 797, 926, 933 nm bands for 488 nm excitation. A detailed description of the results obtained will be given in a separate paper.

Table 2 clearly shows the occurrence frequency of the primary radiation defect: the vacancies (GR1) in both samplings are close, just like the occurrence frequencies of nitrogen-vacancy defects H3 and H4. The occurrence frequency of the NV defect (575 nm) differs by a factor of 2. Significant differences are observed in the occurrence frequencies of the 787 and 933 nm bands, a broad band with a maximum of about 900 nm. The difference in the occurrence frequency of these systems in the AP and RD samplings even exceeds their 45% difference in nitrogen concentration.

DISCUSSION

Our results show the homogeneity of the RD sampling with respect to nitrogen concentration and the fraction of nitrogen in the form of B1 defects. In the distribution of nitrogen defects, the studied AP collection as a whole corresponds to the data of (Fedorova et al., 2013). That publication examined some of the crystals of the AP collections not covered in the present study, as well as 61 RD crystals. Some differences in the results of the data of (Fedorova et al., 2013), especially the number of low-nitrogen crystals, resulted from the fact that the study did not separate the RD and AP crystals, and the distributions were plotted over all samples. Our results show the homogeneity of the RD sampling in the nitrogen concentration distribution, with a maximum at 725 ppm. Only diamonds from the International and XXIII Congress of the CPSU pipes of the Mirny kimberlite field are characterized by the same uniform distribution (Kostrovitsky et al., 2015). For the remaining primary deposits of the Yakutsk diamondiferous province, as well as the Arkhangelsk diamondiferous province (Palazhchenko, 2008; Khachatryan et al., 2008), both the nitrogen concentration and proportion of B1 defects show a greater scatter of values. Ergo the distribution of the nitrogen defect concentration for the RD sampling is similar in character to single (and unique) deposits. In a polygenetic placer deposit with diamonds from several primary sources, it is difficult to expect such a compact distribution of the characteristics. In the AP sampling, up to 45% crystals have a lower nitrogen concentration than in the RD sampling, as well as a significantly larger range of the fraction of B1 defects and, accordingly, model temperatures. Crystals from the alluvial deposits of the Vizhay and Usva rivers are similar to the AP diamonds in their spectroscopic characteristics and morphological features, but they possess an even greater dispersion of characteristics both in the specific features of the PL spectra and the set of nitrogen defects. Significant differences between AP and RD diamond crystals are also observed in the occurrence frequencies of the 787, 933 nm PL bands, a broad band with a maximum of about 900 nm. The nature and features of various luminescence systems is beyond our scope here; this large body of results will be analyzed in detail in a separate paper.

The striking differences between RD crystals and diamonds from the closely located modern placers of the Bolshoi Shchugor and Bolshoi Kolchim river basin cannot be explained by random variations, since the size of the samplings is quite large. When analyzing the possible nature of such differences, of course, one should assume possible subjective bias: the human factor in forming the collections. Enrichment is the only stage at which separation by physical features is possible when forming a collection. The RD crystals were recovered in 1995−1999 using modern separators, and it is rather difficult to assume that 50% of crystals were neglected because their characteristics deviated from the average values for the sampling. Crystals from the APs and Vizhay and Usva rivers were recovered in different years, starting from the 1960s, but their wide range of morphological and spectroscopic features is an indicator of high-quality enrichment.

Differences in the spectroscopic characteristics of RD and AP crystals are supported by the essentially different set of morphological types: in the AP sampling, 12% of crystals have cubic face fragments. Some of these crystals are of type IaA, the second variety according to Orlov's classification; their color is caused by single nitrogen atoms, which are rarely recorded in IR spectra due to their low concentration. Some of the yellow dodecahedroids of type IaA with characteristic luminescence of the S1, S2, S3 systems can be attributed to Orlov's second variety. Some of the crystals with relicts of cubic faces are colorless, systems B1 and B2 were recorded in their IR absorption spectra, and for two crystals, system N3 was recorded in the absorption spectra in the visible range. That is, the morphology of these crystals corresponds to Orlov's second variety, but their physical parameters correspond to the first. The features of this group of crystals require close further study, with the preparation of thin sections, analysis of inclusions, and isotopic studies.

CONCLUSIONS

Diamond crystals of modern alluvial placers of the Bolshoi Shchugor and Bolshoi Kolchim rivers are represented by octahedra, rhombidodecahedra, and cubes: crystals of the first, second, and fourth varieties according to Orlov's classification. In these sources, about 65% are rounded crystals of the Ural (Brazilian) type. In the RD collection, the share of rounded crystals of the Ural (Brazilian) type reaches 84%, while for octahedra and octahedroids, about 9%.

In terms of morphology, nitrogen defect distribution, and PL characteristics, RD and AP crystals show the following differences:

1. AP diamonds bear traces of mechanical wear in the form of (a) edge chipping, (b) crescent-shaped cracks, and (c) etching. No edge chipping or etching was found on RD diamonds; individual crescent-shaped cracks were noted on two crystals. The lozengelike sculpture on RD diamonds is less common and less pronounced.

2. In diamonds of alluvial placers of the Krasnovishersky and Gornozavodsky districts, there are both nitrogen-free crystals of physical type IIa and diamonds of types IaA and IaB1, representing extreme cases on the conditional model temperature scale. In RD diamonds, crystals of physical types IIA, IaA, and IaB1 were not detected.

3. In the PL spectra of AP crystals, the occurrence frequency of the 787, 933 nm bands is 23 and 15%, and in the spectra of RD crystals, 66 and 66%, respectively.

4. Twelve percent of crystals with fragments of cubic faces and dissolution shapes derived from cubes are noted in AP diamonds. In RD diamonds, only one crystal with fragments of cubic faces was found. Some crystals with a cubic habit are of physical type IaB1, and they have PL spectra with a set of various systems: N3, H3, H4, S1, S2, S3. Crystals of this type have not been encountered in other sources; their further detailed study is necessary.

Thus, first, based on the morphological and structural−mineralogical features, crystals of the Rassolninskaya depression differ from crystals of the nearest modern alluvial placers; and second, as a result of the very rare—in terms of their homogeneity—characteristics of the studied samples from the Rassolninskaya depression collection, they can be considered diamonds from one primary deposit.

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