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## MINERALOGICAL FEATURES OF DIAMOND, AMORPHOUS DIAMOND-LIKE CARBON AND GRAPHITE FROM CHAGATAY CARBONATITES (UZBEKISTAN)

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The detailed mineralogical and spectroscopic features of association of different types of native carbon from Chagatay trachyte-carbonatite complex at the northern slopes of the Southern Nuratau Mts (Uzbekistan) are described. The association is presented by the wide list of carbon substances including diamond, graphite, opaque black amorphous carbon, optically transparent amorphous diamond-like carbon, carbon nanofibers and hydrogenated amorphous carbon. The variety of the carbon mineral phases allows suppose non-equilibrium conditions of free carbon formation in Chagatay carbonatites similar to Kymdykol type with fluids participating.

**Introduction.** The new genetic type of diamond connected with carbonatites of Chagatay trachyte-carbonatite complex at the northern slopes of the Southern Nuratau Mts, Uzbekistan was discovered at the end of 20<sup>th</sup> century [5]. By this moment the data about mineralogical particularities of the carbonatitic diamonds are very limited [17] and only one additional carbonatitic diamond-bearing proved object connected with Fuerteventura carbonatites have been found out [23]. The information about diamonds within carbonatites looks discussing for many scientists. Here we report for the first time about detailed mineralogical and spectroscopic features of association of different types of native carbon including numerous optically transparent diamond-like carbon particles, micro-diamond and graphite enriched from the same bulk host carbonatite of Chagatay complex.

**Samples, specimens preparation and methods.** The carbonatites in the Chagatay complex as a rule form connected dikes and rare small stock-like bodies having intrusive contacts with chilling zones [4, 5, 14].

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Among the carbonatites two spatially reproached varieties are distinguished, which are differed in color and mineral composition. According to structural-textural features and mineral content they are devoted to seven varieties. The diamonds were found within the most former melanocratic breccia-like and massive silicate-carbonate carbonatites of biotite-garnet and biotite-pyroxene content which were the object for the carbon mineralization study.

The host free carbon-bearing carbonatites and their minerals preliminary were studied with a complex of petrological and mineralogical methods including wet chemistry, optical microscopy in thin sections and mineral fractions.

Diamond, diamond-like carbon and graphite were enriched from heavy nonmagnetic mineral fraction of a bulk sample of carbonatite (1300 g) by chemical method at the Laboratory of Diamond Mineralogy of the Institute of Geology of Komi Scientific Center of Ural Branch of Russian Academy of Sciences described in detail in [26], amorphous carbon particles and graphite were enriched manually from light mineral fraction after several minutes treatment by 2–3 M HCl for

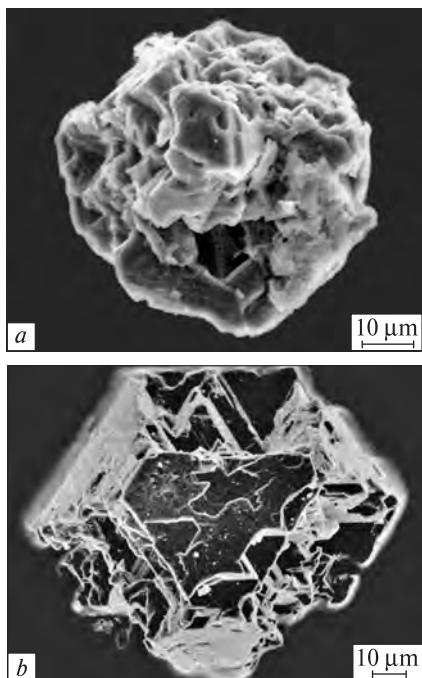


Fig. 1. SEM-image of the skeletal diamond microcrystals: *a* — from Chagatai carbonatites, *b* — from Kumdykol diamond deposit

carbonate removing. Then the particles were picked up under an optical binocular microscope and studied in detail using Raman spectroscopy and electron scanning microscopy in a complex with microprobe analysis.

A scanning electron microscope Jeol JSM-6400 in a complex with microprobe analyzer (Link ISIS-200 and Microspec) was used for mineral chemical composition control and details of morphology.

Raman study was provided with a high resolution Raman spectrometer HORIBA Jobin Yvon HR 800 at room temperature. The 488 and 514 nm of  $\text{Ar}^+$  and 633 nm He-Ne laser excitation with a power 0.2–12 mW was used in dependence of carbon material stability. Analysis was made at room temperature with  $50\times$  and  $100\times$  objectives. The spectral resolution was about  $1\text{ cm}^{-1}$ , the diameter of the analysis spot was about  $1\text{ }\mu\text{m}$ . Spectra were collected in the 100 to  $4000\text{ cm}^{-1}$  range. After background correction, individual peaks were deconvolved using a curve fitting procedure by the combination of Gaussian and Lorentzian functions from software provided by LabSpec 5.36.

**Results. Diamond.** The analyzed microdiamond (Fig. 1) has a skeletal habit and size about  $50\text{ }\mu\text{m}$ , it is optically transparent and almost colorless with brilliant glance on facets. The crystal type has

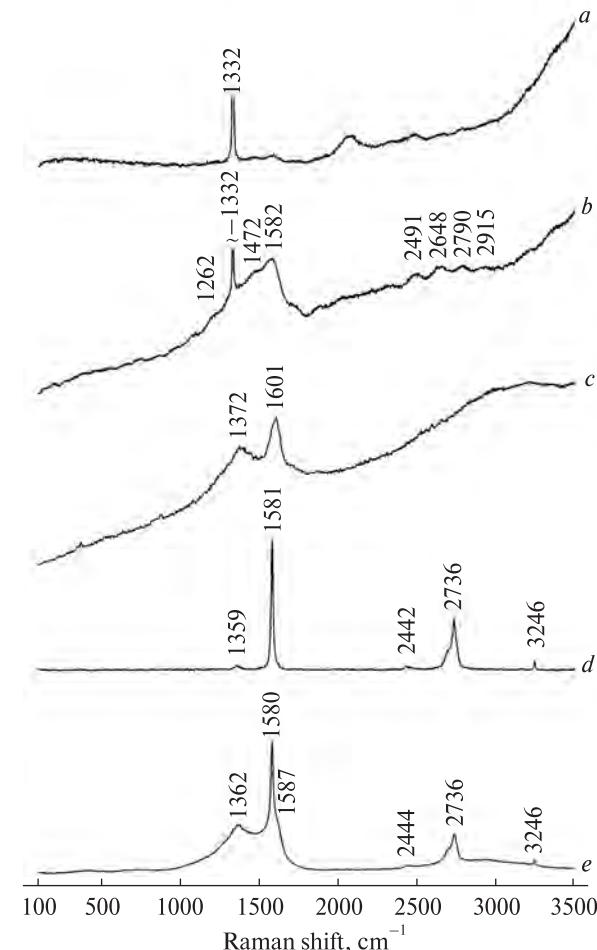


Fig. 2. Raman spectra of the Chagatai microdiamond (*a*), (*b*), amorphous carbon (*c*), high ordered graphite (*d*), amorphous carbon substance with crystalline graphite (*e*). The spectra were received with a Raman spectrometer LabRam HR 800 with 514 nm (*a*–*d*) and 488 nm line (*e*) of external  $\text{Ar}^+$  laser

been described with 13<sup>th</sup> facet variety of octahedron by classification of I.I. Shafranovsky [20]. The analogue morphological type of diamond skeletal crystals was found among microdiamonds of Kumdykol diamond deposit (Kazakhstan) [22].

Three types of spectra from different points of microdiamond surface were identified with Raman spectroscopy. The first type (Fig. 2, *a*) — the spectrum with a strong peak of  $1332\text{ cm}^{-1}$  ( $\text{FWHM} = 4.8\text{ cm}^{-1}$ ), which corresponds to the frequency of diamond lattice —  $T_{2g}$  diamond mode oscillations at room temperature.

The second type of spectrum is additive (Fig. 2, *b*) — it consists of overlapping characteristic bands of different carbon nature. Using decomposition of the Raman spectra by Gaussian-Lorentzian function it was found that the band  $1250\text{ cm}^{-1}$  is corresponding to amorphous  $sp^3$  bonded carbon

[19], the strong peak of  $1332\text{ cm}^{-1}$  is a  $T_{2g}$  diamond mode, the contribution at  $1472\text{ cm}^{-1}$  and the  $G$  band at  $1582\text{ cm}^{-1}$ . For the  $G$  band belonging to Raman active optical vibration  $E_{2g(2)}$  mode [13] it is need to take attention that its full width at half of band maximum (FWHM) is very large and accounts up to  $117\text{ cm}^{-1}$  that corresponds to  $L_a$  around  $1\text{ nm}$  which could be a characteristic rather of amorphous  $sp^2$  carbon. The  $1472\text{ cm}^{-1}$  band (Fig. 2, b) can be interpreted by several ways: belonging either to ta-C : H, diamond-like or polymeric  $a$ -C : H due to  $sp^2$  CH aromatic and  $sp^3$   $\text{CH}_3$  asymmetric or  $sp^3$   $\text{CH}_2$  scissors modes [8].

The third spectrum type characterizes joint presence of crystalline graphite and amorphous carbon on the diamond surface (Fig. 2, e).

Thus, the Raman study of microdiamond crystal from Chagatay carbonatites established the presence of four types of carbon at the same diamond microcrystal surface — high crystalline diamond, amorphous  $sp^3$  bonded carbon, amorphous  $sp^2$  carbon and a mixture of crystalline graphite and amorphous  $sp^2$ .

The used instrumental Raman facility allowed us to register wide spectral region  $450$ – $1100\text{ nm}$ , thus we had possibility to analyze luminescence character of the studied diamond (Fig. 3). According to the received data the skeletal microdiamond has wide region of luminescence  $500$ – $900\text{ nm}$  with the general maximum around  $700\text{ nm}$ . There are two evidently identified quite wide luminescence bands —  $576$  and  $639\text{ nm}$ . The bands good correspond to the typical luminescence centers  $575$  and  $638\text{ nm}$  described by systems of a nitrogen atom impurity with a carbon atom vacancy —  $(\text{N}-V)^0$  and  $(\text{N}-V)^-$  correspondingly [31]. The defects under experimental conditions are usually connected with irradiation influence followed by later temperature treatment under  $900\text{ K}$  and are stable up to  $1400$ – $1500\text{ K}$  [3].

**Graphite.** The graphite of the carbonatites is presented as accessory mineral which is counted up to  $500$  units for  $1\text{ kg}$  of the initial rock mass, with the maxima content up to  $0.05\text{ mas. \%}$ . The graphite has different morphological varieties presented preferably by different aggregates (Fig. 4), xenomorphic and quite good flattened particles of  $0.01$  up to  $2\text{ mm}$  in size.

The first variety has relatively isometric shape of units consisting of subparallel, sputtered sheets and subparallel fiber-like morphological elements, which have sizes around  $1\text{ }\mu\text{m}$  in diameter and up to  $100\text{ }\mu\text{m}$  in prolongation.

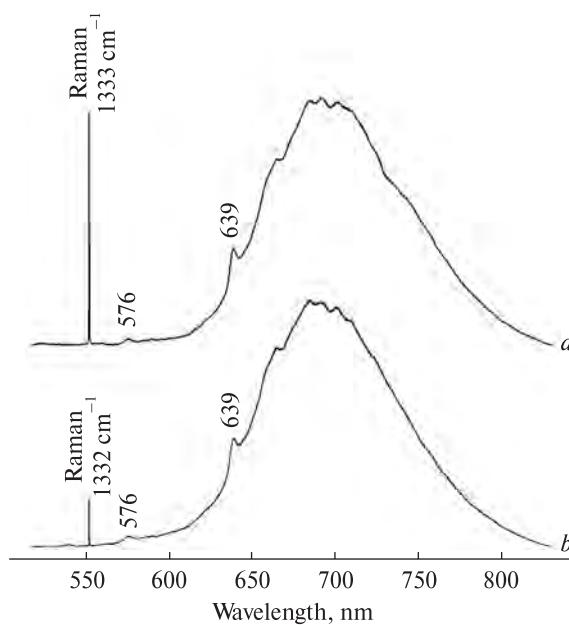


Fig. 3. Luminescence spectra with Raman peaks of the Chagatay microdiamond (a) and Kumdykol diamond (b). Raman spectrometer LabRam HR 800 with  $514\text{ nm}$  line of external  $\text{Ar}^+$  laser was used

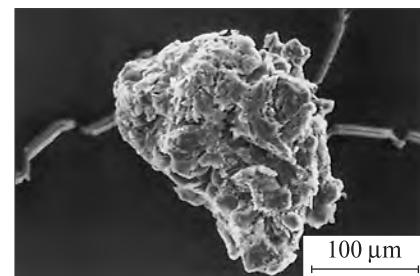


Fig. 4. Graphite aggregate

Xenomorphic graphite exists as hedgehog-like particles up to  $100\text{ }\mu\text{m}$  in size. They do not provide any features of layered texture which presents usually in other genetic graphite types.

The last morphological type is characterized with massive carbon particles which have sizes up to  $100\text{ }\mu\text{m}$ , preferably they are single thick plates with planed shape, sometimes with slightly curved edges and can be surrounded by thin graphite microsheets.

According to Raman spectroscopy (Fig. 2, d) the graphite phase has spectra typical to quite high ordered graphite characterized with intensive sharp  $G$  band of Raman active optical vibration  $E_{2g(2)}$  mode with the position  $1581\text{ cm}^{-1}$  [13]. The band corresponding to the  $D$  disorder induced mode is presented with a very weak band with the central position at  $1359\text{ cm}^{-1}$  which has red shift

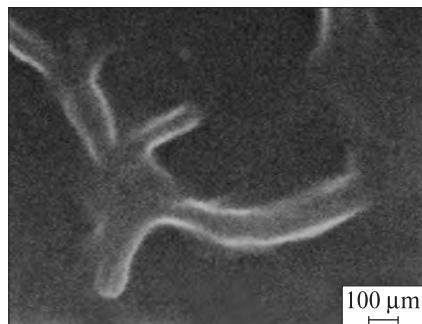


Fig. 5. Carbon nanofibers on a graphite surface

about  $10 \text{ cm}^{-1}$  in compare to normal position for used  $514 \text{ nm Ar}^+$  laser excitation. The spectra are characterized by presence of the well recognized and good structured second order band in the region  $2400\text{--}3300 \text{ cm}^{-1}$ . Thus, the Raman features well correspond to high ordered crystalline graphite [13, 28, 32]. Calculated  $L_a$  parameter gets up to  $130 \text{ nm}$  using the method of F. Tuinstra and J.L. Koenig [29].

Within tight bulk aggregates with high ordered graphite carbon nanofibers were found (Fig. 5) which were described some earlier in detail [27].

**Amorphous carbon.** Amorphous carbon ( $a$ -carbon) substance was found on the microdiamond surface and as xenomorphic opaque black aggregates with sizes up to  $0.1 \text{ mm}$ . The carbon aggregates including opaque and transparent fragments were studied in detail with Raman spectroscopy within bulk aggregates and as disintegrated particles for correct spectra analysis of independent particles.

$A$ -carbon on a diamond surface is presented by amorphous matter which had very wide two bands centered at  $1372$  and  $1601 \text{ cm}^{-1}$  with FWHM counting  $310$  and  $83 \text{ cm}^{-1}$  correspondingly (Fig. 2, c). The spectra were characterized with almost full absence of the second order band and existing of quite intensive luminescence in the

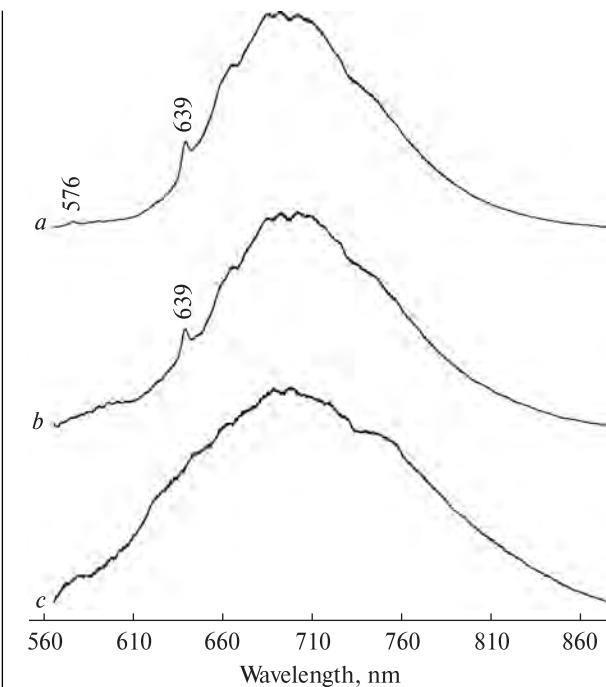


Fig. 6. Luminescence spectra: microdiamond (a), microdiamond with amorphous carbon substance (b) and amorphous carbon substance (c). The spectra were received with a Raman spectrometer LabRam HR 800 with  $514 \text{ nm}$  line of external  $\text{Ar}^+$  laser at room temperature

region  $550\text{--}900 \text{ nm}$  with the maxima around  $700 \text{ nm}$ .

To identify the carbon substance we have provided Raman data comparison with the varieties of poor ordered carbons such as diamond-like carbon, nanocrystalline graphite, glass-like carbon, soot, onion-like carbon and fully amorphous carbon. But among numerous published and our own experimental Raman data we have not found an absolutely full analogue.

Taking into attention the bands positions and their FWHM we have supposed that the studied carbon was presented rather by essentially amor-

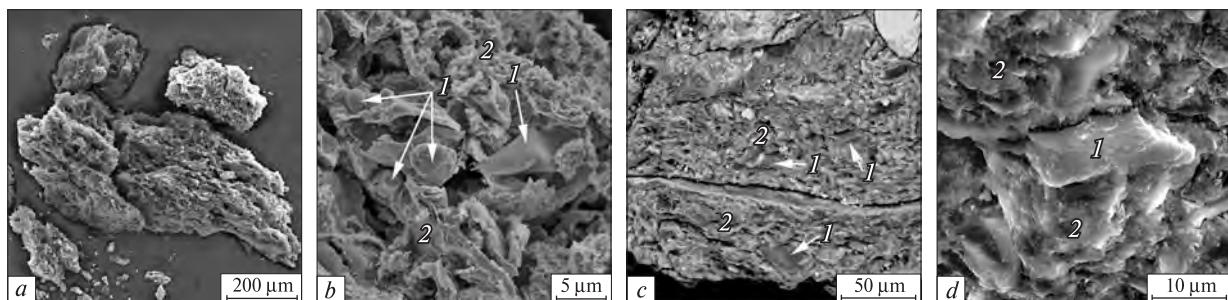


Fig. 7. Aggregates of amorphous carbon: a — friable aggregates, b — magnified fragment of friable aggregate with inclusions of optically transparent  $a$ -carbon (in a center — 1) surrounded by opaque matter — 2; c — massive aggregate with inclusions of optically transparent  $a$ -carbon (in a center — 1) surrounded by opaque matter — 2; d — increased fragment (from 7, c)

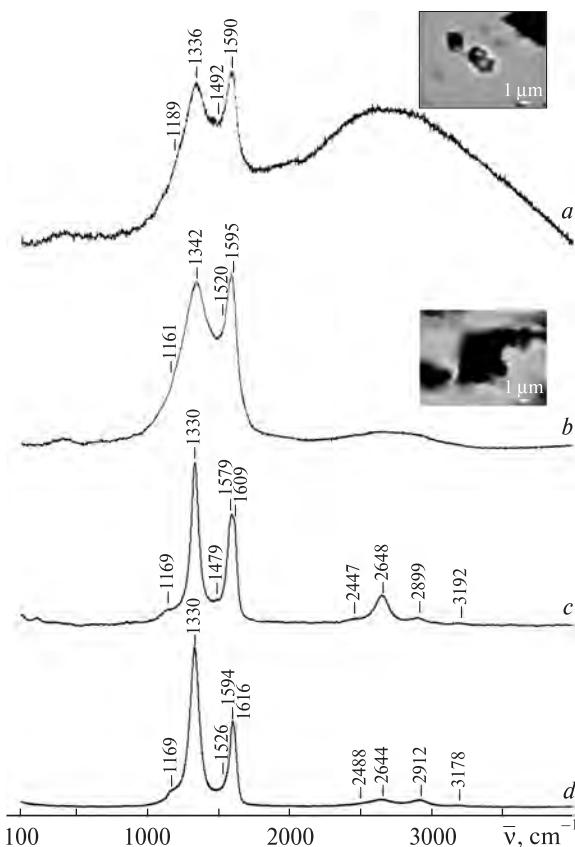


Fig. 8. Raman spectra: *a* — optically transparent *a*-carbon and co-followed photo in transparent light; *b* — opaque *a*-carbon and co-followed photo in transparent light; *c* — synthetic glass-like carbon SU2000; *d* — shungite

phous carbon with high content of  $sp^3$  C—H band at  $1375\text{ cm}^{-1}$  and  $sp^2$  C=C bonds at  $1603\text{ cm}^{-1}$  [19, 33]. The last band could be a characteristic of nanocrystalline graphite [9], but in this case the FWHM of the *G* band have to be essentially smaller — about  $40\text{ cm}^{-1}$  with the position about  $1585\text{ cm}^{-1}$  and a shoulder at  $1620\text{ cm}^{-1}$  which is well divided using high resolution spectrometer. Additionally the spectrum should has essentially smaller Raman shift for *D* band (around  $1350\text{ cm}^{-1}$  for used laser excitation) and good recognized and structured second order [26]. By this reasons we

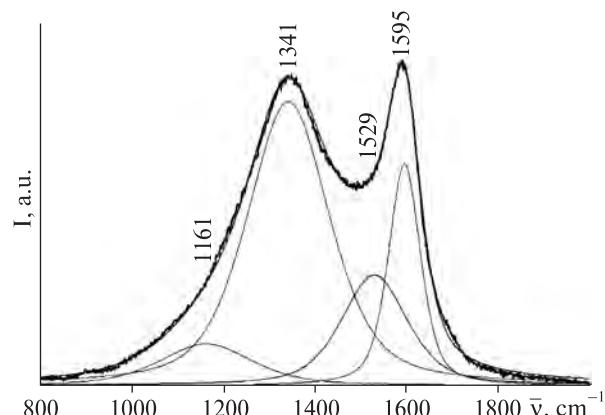


Fig. 9. Raman spectrum of *a*-carbon deconvoluted by fitting procedure with Gaussian and Lorentzian functions

have concluded that the matter cannot be nanocrystalline graphite and is characterized by amorphous nature of the carbon substance presented by a mixture of carbon with  $sp^3$  C—H and  $sp^2$  C=C bonds.

The presence of mentioned above luminescence which has some analogues features with the Chagatai diamond (described below) (Fig. 6) allows to suppose about presence of some nanodiamond clusters with  $sp^3$ -carbon within the amorphous matter.

Opaque black amorphous carbon from micro-grain aggregates (Fig. 7) is characterized by more primitive spectrum (Fig. 8, *b*; 9) presented by two general wide bands perhaps corresponding to *D* and *G* bands pointed at  $1340$  and  $1595\text{ cm}^{-1}$  correspondently and third component around  $1530\text{ cm}^{-1}$  with absence of the second order. For comparison with other low ordered carbon substances we demonstrate spectra of glass-like carbon and shungite (Fig. 8, *c*, *d*; Table 1). It is evidently that the describing opaque black *a*-carbon has essentially wider FWHM of *D* and *G* bands and absence of the structured second order band around  $2500$ – $3000\text{ cm}^{-1}$  which differ the analyzed carbon from the control specimens and characterize it as amorphous carbon matter. The

Table 1. Raman features of Chagatay *a*-carbon and control of low ordered specimens,  $\text{cm}^{-1}$

Specimen	Band									
	<i>D</i> 2		<i>D</i>		<i>D</i> 3		<i>G</i>		<i>D</i> 4	
	Position	FWHM	Position	FWHM	Position	FWHM	Position	FWHM	Position	FWHM
Optically transparent <i>a</i> -carbon	1189	169	1336	230	1492	150	1590	100	—	—
Opaque <i>a</i> -carbon	1161	306	1342	220	1520	155	1595	96	—	—
Glass-like carbon SU-2000	1138	172	1332	69	1479	100	1579	73	1609	54
Shungite	1169	87	1330	80	1526	100	1594	53	1616	32

**Table 2. Raman spectroscopy and luminescence data on Chagatay and Kumdykol skeletal diamonds**

Diamond sample description	Raman spectroscopy data, $\text{cm}^{-1}$		Luminescence data, nm			
	Peak position	FWHM	Band position	FWHM	Band position	FWHM
Chagatay, 955-1	1332	4.8	576	6	639	8
Kumdykol, d0-b4	1332	4.5	576	8	639	8

additional shoulder band at  $1160 \text{ cm}^{-1}$  can be related to disordered or microcrystalline  $sp^3$  carbon (similar to  $D$  band for graphite) [34].

Optically transparent amorphous diamond-like carbon. Micrometer-sized optically transparent amorphous carbon inclusions were found as inclusions within the mentioned aggregates of black nontransparent amorphous carbon, described above. Raman study was provided for independent particles which were separated by manual crushing of the host aggregates of opaque black amorphous carbon. Morphologically transparent  $a$ -carbon is presented by noncrystalline particles (Fig. 7) usually the details are not visible at optical microscopy observations, but sometimes for larger particles sub-crystalline cubic or octahedral habit were recognizable (Fig. 8, a).

The typical spectrum of the studied optically transparent carbon can be decomposed to bands similar to opaque  $a$ -carbon (Fig. 8; Table 1). Some difference in the positions cannot be informative as they have quite large mistake at such large value of FWHM, except  $D3$  band which essentially blue shifted for about  $30 \text{ cm}^{-1}$  and could be useful. It looks that the informative feature in the spectrum is presented by an intensive wide luminescence band which is always absent in the opaque  $a$ -carbon. Among known carbon substances there are no exactly similar spectra. It is possible to think that the transparent  $a$ -carbon could be similar to recently described optically transparent hard carbon from impact astroblems presented by El Goresy et al. [6, 7]. At the same time the authors supposed about Raman-inactive character of the phase. Taking into attention that in our case we analyzed independent particles which evidently demonstrate Raman spectrum we sure that the spectrum is its own Raman feature.

By the detail analysis of Raman data of transparent and opaque  $a$ -carbons we have found that they have equal relation of integral intensities  $I_D/I_G$  (2.7) and essential difference in  $I_D/I_{D3}$  corresponding to 0.17 and 0.34 respectively. The last can be a result of better ordering in transparent matter with possible larger input of  $sp^3$  [11, 19]. The

Raman spectroscopy features allow suppose, that the optically transparent carbon matter could be amorphous diamond-like substance.

**Discussion.** By this moment there are two principle different hypotheses of diamond formation of the Chagatay carbonatites. The first is connected with magmatic stage of carbonatites formation at subvolcanic facie of the Chagatay trachyte-carbonatite complex [4, 5]. The second point supposes that the diamond-bearing rocks of the complex have been formed at collision crustal zone and the diamonds have been crystallized under anomalous tectonic pressures and high temperatures from deep fluids similar to Kumdykol diamond deposit type [14].

The principle possibility of diamond formation directly at magmatic stage of carbonatites formation is supported earlier by experimental diamond synthesis [1, 2, 16, 17]; Liu et al., 2001; Yamaoka et al., 2006 et al.) and diamond finding within the real magmatic carbonatites of Fuerteventura Island [23].

According to experimental data the carbonatite melts are fruit-full for diamond crystallization either with added of some free carbon source into the crystallization system [2, 16, 17]; by  $\text{FeCO}_3$  and  $\text{MnCO}_3$  decarbonisation (Liu et al., 2001),  $\text{CaCO}_3$  decarbonisation with reduced C—O—H fluid presence at HP—HT (Yamaoka et al., 2006) or with melted geochemically natural carbonatite system [1, 26]. The conditions of diamond formation were within the frames of pressure 4—13 GPa and 1500—2000 K.

The ways provide formation of free carbon with specific morphological and structural features. The study of the high pressure high temperature synthesis products demonstrated essentially different data in compare to Chagatay carbon phases. The synthetic products were presented by diamonds of bulk octahedral habit of single crystals and their growths and twins with different ordering and were followed by glass-like carbon and high crystalline graphite [10, 16, 24—26]. The natural diamond find within Canarian diamonds have the same morphological features [23].

The described skeletal Chagatay diamond has no any full morphological analogues among synthetic or natural crystals except Kumdykol diamonds from metamorphic rocks [15, 22], the last has the 13<sup>th</sup> facet variety of octahedron after I.I. Shafranovsky [20] as one of the most specific and abundant type.

We have provided compare analysis of the skeletal Chagatay diamond with other features of Kumdykol diamonds of the same morphological type and co-following free carbons. As a result it have been found out that their structural particularities are essentially similar, such as a level of the general diamond matter ordering (Table 2) and defects providing the same luminescence (Fig. 3). In spite of the both diamond types were enriched with thermochemical method the pointed luminescence centers could not be manually stimulated as it was mentioned above the defects should be received under essentially higher temperatures and should be stable up to 1400 K.

In addition it is important to take attention that in difference to analyzed Chagatay variety the kimberlite diamonds usually have essentially better ordering and luminescence [3, 30].

Among the co-following free carbon phases of the Chagatay diamond type we did not find glass-like carbon which would be the best direct indicator of free carbon formation from high pressure high temperature carbonate melt as it was reported above. As for high crystalline graphite it is similar by the ordering to any other well crystalline graphite.

The other important point is following from the less ordered Chagatay carbon substance which is presented within the skeletal diamond and as amorphous carbon inclusions in carbon aggregates. The matter is characterized perhaps with amorphous diamond-like carbon of different  $sp^3$  and  $sp^2$  content: optically transparent — high  $sp^3$  carbon containing and black opaque — low  $sp^3$  carbon containing. The  $sp^3$  and  $sp^2$  carbon probably exist as tiny nanosized clusters. Such carbon states were not known for traditional diamond deposits. By the other hand Kumdykol skeletal diamonds have similar structural features and luminescence (Fig. 4; Table 1).

The solid amorphous hydrogen-contain carbon has especial interest and described for the first time in such type of objects. The presence of the phase together with moissanite can directly point to the high residual environment of the host dia-

mond-containing rock at list around local points. In addition this should be an indicator of hydrocarbons participation in free carbons formation.

Among the variety of carbon matter in Chagatay diamond-bearing carbonatites it is need to take attention to graphite — carbon nanofibers bulk aggregates which were just described [27]. The aggregates rather support possibility of hydrocarbons gas participation in free carbon/diamond formation. Similar carbon nanofibers were met earlier within Kumdykol diamonds as similar bulk aggregates [21].

Summarizing the listed above typomorphic features of the Chagatay diamond type and the co-following carbon phases we suppose that the diamond formation of the Chagatay carbonatites should not be as a result of direct magmatic process. Investigated in this work carbonatites had essential recrystallization with active fluid treatment. According to a complex of the received data diamond formation in Chagatay complex rather should be similar to Kumdykol diamond type with active fluid (probably of deep origin) participation at the process of diamond crystallization. The point is in a good agreement with the absence of usual diamond mantle indicators within the host Chagatay carbonatites [14]. The graphite presence with the described typomorphic features can be an indicator of diamond presence in carbonatites [12, 18].

**Conclusion.** The study of carbon mineralization from Chagatay carbonatites allow to describe the wide list of carbon substances in association — diamond, graphite, opaque black amorphous carbon, optically transparent amorphous diamond-like carbon, carbon nanofibers and hydrogenated amorphous carbon which supports non-equilibrium conditions of free carbon formation.

The provided study is very important for understanding of formation mechanism of the Chagatay diamond-containing object. According to the received data it is following that the microdiamonds crystallization of the Chagatay carbonatites probably are not connected with magmatic process directly. They rather should be formed within the Earth crust with active reduced fluid participation (probably of deep fluid origin) similar to Kumdykol diamond type.

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**МИНЕРАЛОГИЧЕСКИЕ ОСОБЕННОСТИ  
АЛМАЗА, АМОРФНОГО АЛМАЗОПОДОБНОГО  
УГЛЕРОДА И ГРАФИТА ИЗ ЧАГАТАЙСКИХ  
КАРБОНАТИТОВ (УЗБЕКИСТАН)**

Детально описаны минералогические и спектроскопические особенности ассоциации различных типов углерода из Чагатайского трахит-карбонатитового комплекса северной части Южного Нуратау (Узбекистан). Ассоциация представлена широким спектром углеродных веществ, включая алмаз, графит, не-

прозрачный черный углерод, оптически прозрачный аморфный алмазоподобный углерод, углеродные новолокна и гидрогенезированный аморфный углерод. Разнообразие углеродных фаз позволяет предположить неравновесные условия формирования свободного углерода в чагатайских карбонатитах аналогично кумдыкольскому типу с участием флюидов.

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**МІНЕРАЛОГІЧНІ ОСОБЛИВОСТІ АЛМАЗУ,  
АМОРФНОГО АЛМАЗОПОДІБНОГО  
ВУГЛЕЦЮ І ГРАФІТУ З ЧАГАТАЙСЬКИХ  
КАРБОНАТИТІВ (УЗБЕКИСТАН)**

Детально описано мінералогічні та спектроскопічні особливості асоціації різних типів вуглецю з Чагатайського трахіт-карбонатитового комплексу північної частини Південного Нуратау (Узбекистан). Асоціація представлена широким спектром вуглецевих речовин, що включають алмаз, графіт, непрозорий чорний вуглець, оптично прозорий аморфний алмазоподібний вуглець, вуглецеві новолокна і гідрогенізований аморфний вуглець. Різноманітність вуглецевих фаз дозволяє припустити нерівноважні умови формування вільного вуглецю в чагатайських карбонатитах аналогічно кумдикольському типу з участю флюїдів.