A series of yellow cuboid diamonds from alluvial placers in the north-eastern Siberian platform have been examined with Fourier-transform infrared (FTIR) absorption spectroscopy, photoluminescence (PL) and electron paramagnetic resonance (EPR). All crystals having characteristic FTIR spectra with predominantly C and A centres belong to mixed type IaA/Ib with small total nitrogen content (up to 330 ppm). PL is characterized by many bands with two bands related to the NV
 superscript 0 and NV
 superscript – centres common to all the samples. The FTIR spectra are also characterized by deformation-induced ‘amber’ centres. Some of the yellow diamonds studied show new bands at 4272, 4548, and 5960 cm
 superset –1 instead of the bands at 4060 and 4170 cm
 superset –1 common for ‘amber’ centres. An EPR study has revealed the presence of OK1 centres associated with titanium impurity. The plastic deformation of the studied diamonds is expressed as a ‘tatami’ strain pattern in the features of their internal structure.

Keywords: diamond, cuboid, defects, nitrogen, Siberian platform

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1. Introduction

The Siberian Platform hosts more than one thousand known kimberlite pipes. Hundreds of kimberlite pipes have been discovered in the north-eastern region of the Siberian platform, most of them are non-diamondiferous or extremely poor in diamonds. However, nearly 70% of the diamond-bearing alluvial placers occur here (Grakhanov et al. 2007). The primary sources of the diamonds in these placers have not been discovered yet despite intense exploration. According to the mineralogical classification system of Orlov (1977), the alluvial placer diamonds may be divided into the following three groups: (1) typical octahedral-to-rounded diamonds of variety I; (2) yellow-orange or dark grey cuboids of varieties II and III; and (3) rounded dark crystals of variety V. Minerals of eclogitic parageneses with a high content of titanium compounds dominate the available data regarding the composition of mineral inclusions in diamonds from placers (Sobolev et al. 1999; Shatsky et al. 2014; 2015).

Cubic-shaped diamond crystals (cuboids), forming a continuous color series from yellowish-green to yellow and orange, are assigned to variety II (Orlov 1977). Diamonds of this variety comprise approximately 7.5% of the population of diamonds from alluvial placers (Grakhanov et al. 2007) but are rarely found in kimberlite pipes. Yellow cuboid diamonds attract the attention of many researchers because of their specific spectroscopic and isotopic characteristics (Nadolinny et al. 2009b; 2012; 2015; Hainschwang et al. 2012; 2013; Mineeva et al. 2013; Zudina et al. 2013; Titkov et al. 2015b; Zedgenizov et al. 2016; 2017; Reutsky et al. 2017; Smit et al. 2018; Vasilev et al. 2020). This study is part of continuing research on alluvial diamonds from the north-eastern Siberian platform. We provide a new dataset on structural defects in yellow cuboid diamonds as revealed by FTIR absorption spectroscopy, photoluminescence (PL) and electron paramagnetic resonance (EPR).

2. Samples and experimental methods

Eleven diamonds coloured from pale yellow to dark orange-brown were selected from a commercial diamond collection from alluvial placers in the north-eastern Siberian platform. The diamonds presented as transparent cuboid (i.e. nearly cubic shape) to subrounded crystals 2–3 mm in size (Fig. 1). Most of them are significantly resorbed and have a typical tetrahedroid morphology. All samples were cut and polished into thin plates to observe their internal structure and acquire the IR spectra from different growth zones. The birefringence
Fig. 1 Yellow cuboid diamonds from alluvial placers of the northeastern Siberian platform, showing a range of colors and morphologies: left column – reflected light images of original crystals 1–11; middle column – transmitted light images of polished plates; right column – birefringence images.
patterns of the polished plates were observed using a Zeiss Axioskeop 40 microscope. The infrared spectra of the diamonds were recorded using a Bruker VERTEX 70 FTIR spectrometer equipped with a HYPERION 2000 microscope. Spectra were measured in a range of 7000–600 cm⁻¹, with 32 scans at a resolution of 2 cm⁻¹. The spectra were recorded using an aperture of 60 × 60 μm. FTIR spectra were recorded at suitable points for all samples before polishing. The thin plates were then cut and the number of nitrogen centres has been estimated from the spectra recorded from different growth zones of each crystal. After the baseline subtraction and standard normalization to the intrinsic absorption of diamonds (12.8 cm⁻¹ at 2030 cm⁻¹, Zaitsev 2001), the concentrations of the major nitrogen defects (C – single-substitutional nitrogen atoms, A – adjacent-substitutional pair of nitrogen atoms, B – four nitrogen atoms surrounding a vacancy, and X – positively charged single-substitutional nitrogen, N⁺) were calculated by the decomposition of the one-phonon region of the IR spectra into individual components based on the ratios \( \left[ N_A \text{ (ppm)} = 16.5 \times \alpha_{1282} \text{ (cm}^{-1}\right); \quad N_\text{C} \text{ (ppm)} = 79.4 \times \alpha_{1282} \text{ (cm}^{-1}\right); \quad N_\text{B} \text{ (ppm)} = 25 \times \alpha_{1130} \text{ (cm}^{-1}\right); \quad \text{and } N_X = 5.5 \times \alpha_{1332} \text{ (cm}^{-1}\right) \) proposed by Kiflawi et al. (1994), Boyd et al. (1995), Lawson et al. (1998), and Zaitsev (2001).

Photoluminescence (PL) spectra in the range 400–850 nm at 80 K were acquired under the 313 nm line (filtered through UFS-2 glass) of a mercury DRT-230 lamp and diode laser with \( \lambda = 532 \text{ nm} \) for the range 540–850 nm using a DFS-24 spectrometer equipped with a double-grating monochromator with 1200 lines/mm gratings and an FEU 79 photomultiplier for signal detection. The spectral resolution varied from 0.3 to 0.6 nm depending on the luminescence intensity and half-width of the zero-phonon lines (ZPL). No correction for the response of the recording system was made.

EPR spectra were measured at room temperature on a Radiopan SE/X 2543 spectrometer with a built-in NMR magnetometer. The sample was fastened on a custom-built goniometer allowing two-axis rotation of the crystal in the cavity. The output EPR signal was digitized with a custom 16-bits analogue-digital converter interfaced with a PC. The 100 kHz modulation amplitude was selected so that narrow EPR lines without significant broadening were detected. The microwave power was reduced to 0.6 mW when the spectra of defect centres were collected because of EPR signal saturation.

3. Results

3.1. FTIR data

The FTIR spectra of the original diamond crystals are displayed in Fig. 2. The diamonds studied contain predominantly C (single nitrogen atom; Kiflawi et al. 1994) and A (pair of neighbouring nitrogen atoms; Davies 1976) centres and may be assigned to mixed Type IaA/Ib. Total nitrogen content is low from 46 to 330 ppm (Tab. 1). For diamonds with a combination of nitrogen defects, the content in each form is determined by computer minimization of the component spectra to the experimental spectrum. The diamonds studied can be divided into two groups. The first group contains only two samples (2 and 6) which show IR spectra that may be fitted using mostly A and C defects with the possible addition of a minor amount of B (a group of four nitrogen atoms replacing carbon around the vacancy; Zaitsev 2001) and X centres (Fig. 3). These samples have higher total nitrogen content.

Fig. 2 The one-phonon region of FTIR spectra for raw diamonds. The numbers of the samples are marked.

Fig. 3 Curve fitting analysis of the one-phonon region of FTIR spectrum for sample 2 (experimental black curve). The decomposition reveals the presence of the A (blue), C (red), B (dark cyan)) and X (magenta) nitrogen defects. The sum of those components is the fitting curve (short dot dark yellow line).
The second group is characterized by low total nitrogen content wherein the number of C defects is more than or comparable to the number of A defects. Besides A and C centres they also show the presence of specific X and Y centres (Figs. 4 and 5). The X centre with a sharp IR peak at 1332 cm\(^{-1}\) has been related to a positively charged single substitutional nitrogen atom (Lawson et al. 1998). The Y centre with the main band at 1140–1150 cm\(^{-1}\) has been described in Type Ib and IaA/Ib of diamonds (Hainschwang et al. 2012; Titkov et al. 2015b; Tab. 1)

**Tab. 1** Infrared (FTIR), electron paramagnetic resonance (EPR) and photoluminescence (PL) characteristics of yellow cuboid diamonds from alluvial placers of the northeastern Siberian platform

<table>
<thead>
<tr>
<th>No</th>
<th>Color</th>
<th>FTIR data</th>
<th>EPR</th>
<th>PL centers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>C, ppm</td>
<td>A, ppm</td>
<td>X, ppm</td>
</tr>
<tr>
<td>1</td>
<td>y–or</td>
<td>112</td>
<td>17</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>y–green</td>
<td>36 72</td>
<td>13 13</td>
<td>3 3</td>
</tr>
<tr>
<td>3</td>
<td>grey–y</td>
<td>51</td>
<td>172</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>grey</td>
<td>50</td>
<td>26</td>
<td>2</td>
</tr>
<tr>
<td>5</td>
<td>or–br</td>
<td>28</td>
<td>18</td>
<td>1</td>
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<tr>
<td>6</td>
<td>y</td>
<td>13 26</td>
<td>28 23</td>
<td>1 1</td>
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<tr>
<td>7</td>
<td>br–red</td>
<td>59</td>
<td>24</td>
<td>2</td>
</tr>
<tr>
<td>8</td>
<td>br–red</td>
<td>54 62</td>
<td>23 27</td>
<td>2 2</td>
</tr>
<tr>
<td>9</td>
<td>grey–red</td>
<td>47</td>
<td>172</td>
<td>8</td>
</tr>
<tr>
<td>10</td>
<td>y–green</td>
<td>50 43</td>
<td>160 299</td>
<td>5 4</td>
</tr>
<tr>
<td>11</td>
<td>y–or</td>
<td>62</td>
<td>29</td>
<td>5</td>
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</table>

y – yellow, br – brown, or – orange, C – central zone, R1; 2 – opposite outer zones
Specific spectroscopic features of yellow cuboid diamonds

1. Massi et al. (2005) have indicated that there are at least four different configurations of ‘amber’ centres, and all of them have been found in crystals showing the lamellae of plastic deformation and nitrogen in A-form. The spectra of yellow cuboid diamonds appear to be made up of a superposition of independent bands at 4067, 4098, 4113, 4137, 4170, and possibly 4676 cm$^{-1}$ that cannot be assigned to any groups proposed earlier (Massi et al. 2005). Usually, for such diamonds, the bands at about 4060 and 4170 cm$^{-1}$ are observed at room temperature (Fedorova et al. 2013; Massi et al. 2005; Titkov et al. 2015b). Titkov et al. (2015b) have observed additional bands at 3460 and 4244 cm$^{-1}$. We also observed the bands at 3468 and 4234 cm$^{-1}$ in the yellow cuboid diamonds. New bands at 4272, 4548, and 5960 cm$^{-1}$ are also observed in sample 5 (Fig. 5).

3.2. EPR data

The paramagnetic centre P1 related to the substitutional nitrogen atoms is observed in EPR spectra of all the yellow cuboid diamonds studied in this work. The number of paramagnetic nitrogen defects may be evaluated according to the method described by van Wyk et al. (1997). Peak-to-peak line widths changing from 0.8 G in sample 4 to 1.2 G in sample 8 correspond to P1 concentrations between 60 and 120 ppm (van Wyk et al. 1997). The minimal and maximal concentrations of C centres determined from FTIR spectra generally corresponded to EPR measurements (Tab. 1). Each method has advantages. The IR beam penetrates through the entire thickness of the crystal or plate and measuring in one place necessarily includes other

Zedgenizov et al. 2016). A broadened C centre absorption indicates the Y centre shifted to a position between 1135 and 1140 cm$^{-1}$, instead of the standard position at 1130 cm$^{-1}$. Additional peaks at 1353, 1358, 1363, 1374 and 1387 cm$^{-1}$ also appear to be associated with a Y centre (Hainschwang et al. 2012; Titkov et al. 2015b). Recently, the peak intensity at 1358 cm$^{-1}$ and the peak at 1332 cm$^{-1}$ were correlated and hence the latter peak is involved in the full spectrum of Y centre (Reutsky et al. 2017). A minor amount of Y centres might be present in the diamonds of the first group instead of B centres as suggested (see Fig. 3).

The peak at 3107 cm$^{-1}$ related to C–H vibrations is detected in most of the samples studied. Based on recent experimental data and theoretical calculations it was shown that the VN$_3$H structural unit is the most appropriate candidate for the 3107 cm$^{-1}$ centre (Goss et al. 2014). The diamonds show low-intensity absorption at 3107 cm$^{-1}$ (Tab. 1) and rare additional weak H-related features (peaks at 2854, 2870, 2932, 2973, 3144, 3309, 3343, 3394 cm$^{-1}$), in contrast to some samples with intense Y centre where the hydrogen-related features comprised a large number of intense, sharp peaks (Hainschwang et al. 2012).

Another specific feature of yellow cuboid diamonds is absorption bands in the near-infrared region (between 3900 and 6000 cm$^{-1}$) (Tab. 1), associated with so-called ‘amber’ centres (DuPreez 1965; Massi et al. 2005). These bands in four studied samples are shown in Fig. 6. The ‘amber’ centres have been detected in many natural diamonds with global origins and are typically assigned to plastic deformation (DuPreez 1965; Massi et al. 2005; Fedorova et al. 2013; Hainschwang et al. 2013; Titkov et al. 2015b). Massi et al. (2005) have indicated that there are at least four different configurations of ‘amber’ centres, and all of them have been found in crystals showing the lamellae of plastic deformation and nitrogen in A-form. The spectra of yellow cuboid diamonds appear to be made up of a superposition of independent bands at 4067, 4098, 4113, 4137, 4170, and possibly 4676 cm$^{-1}$ that cannot be assigned to any groups proposed earlier (Massi et al. 2005). Usually, for such diamonds, the bands at about 4060 and 4170 cm$^{-1}$ are observed at room temperature (Fedorova et al. 2013; Massi et al. 2005; Titkov et al. 2015b). Titkov et al. (2015b) have observed additional bands at 3460 and 4244 cm$^{-1}$. We also observed the bands at 3468 and 4234 cm$^{-1}$ in the yellow cuboid diamonds. New bands at 4272, 4548, and 5960 cm$^{-1}$ are also observed in sample 5 (Fig. 5).
zones. In the samples with unknown IR absorption attributed to Y centres, the simulation of the experimental FTIR spectrum is uncertain (see Fig. 4). EPR can only measure the concentration of the whole crystal, which is estimated roughly by the method of van Wyk et al. (1997). Another possible method of EPR evaluation of P1 content is by comparison with the standard sample. Comparison gave a lower concentration versus FTIR-based measurement of the C defects (Mineeva et al. 2013; Titkov et al. 2015a).

In most samples, the OK1 centre is also fixed by EPR with concentrations of about two orders less than those of the P1 centres (for example, in sample 6 the OK1 content is about 1 ppm) (Fig. 7). The OK1 centre was first detected by Klingsporn et al. (1970). Based on electron-nuclear double resonance (ENDOR), the model of the chain C–N–C–O–C in the [110] direction was suggested for this centre (Newton and Baker 1989).

3.3. Photoluminescence

3.3.1. 313 nm excited PL

When excited at 313 nm the crystals had yellow or yellowish-green luminescence of different intensities. A zonal luminescence pattern was distinctly observable. In addition to the centre S1 (ZPLs at 503.4 nm and 510.7 nm), the centre H3 (with an N–V–N structural model) was recorded nearby at 503.2 nm. This proximity made it difficult to distinguish them due to their weak intensities. The 415 nm centre (N3 – three nitrogen atoms plus a vacancy, N3V) has been registered in three diamonds at lower intensities. In sample 7, a weak S1 centre was observed on a broad background, and three samples did not show any luminescence at this excitation.

3.3.2. 532 nm excited PL

When excited at 532 nm, the crystals had a bright orange luminescence. This colour of luminescence is caused by the presence of either neutral (NV0) or negatively charged (NV-) nitrogen-vacancy centres with ZPL at 575 and 637 nm, respectively (Fig. 8a, b). The 612.4 nm centre (the 612.5 or 613 nm centre in some studies) was observed in nine samples with the highest intensity of the ZPL at 612.4 nm in sample 6. The vibrational sidebands are formed by interactions with 38 and 78 meV phonons close to a quasi-local vibration of a vacancy. A peak at 612 nm has been observed in untreated plastically deformed brown and pink diamonds (Hainschwang et al. 2006; Titkov et al. 2008; Tretiakova 2009; Gaillou et al. 2010; Yuryeva et al. 2020). The 613-nm centre has also been observed in the grains of polycrystalline samples, from colourless to those dyed various shades of grey and yellow (Yang et al. 2012). We did not find evidence of a
relationship between the intensity of the 613 nm centre and the total nitrogen concentration. It can, therefore, only be assumed that there are nitrogen atoms and vacancies in its structure. The 613 nm centre is quite stable at high temperatures and can be completely annealed only at 2000 °C and 6.5 GPa (Tretiakova 2009; Yuryeva et al. 2015).

The centre with ZPL at 635.7 nm and local vibration at about 37 meV was observed in the spectra of five of the diamonds studied. This centre has previously been detected in many yellow cuboid diamonds (Zudina et al. 2013; Zedgenizov et al. 2016). The correlation between the 635.7 nm luminescence system and the N3 centre detected by EPR has recently been demonstrated by Nadolinny et al. (2015). However, although we observed the 635.7 nm system in yellow cuboid diamonds we did not detect the N3 centre with EPR. A set of other centres was also observed, i.e. in sample 7 the highest peak was seen at 635.7 nm (Fig. 8b, Tab. 1). For sample 7 some Ni-related lines were observed at 596.0, 599.0 and 603.5 nm in PL spectra (Yelissyeiev and Kanda 2007; Tretiakova 2009; Dobrinets et al. 2013).

4. Discussion

Yellow cuboid diamonds from the alluvial placers of the north-eastern Siberian platform are of particular scientific interest. Previously described characteristics of such diamonds i.e., primary cuboid morphology, light carbon isotope composition, and low aggregated nitrogen (Type Ib has a specific yellow colouration), suggest an unusual primary source (Zedgenizov et al. 2016). FTIR studies of similar diamonds have revealed a specific set of C, A, X and Y centres (Titkov et al. 2015b). In nine of the diamonds studied we detected the same set of centres. Only two samples (2 and 6) have negligible amounts of Y centres present. The presence of C centres and absence of B centres are usually attributed either to relatively cool conditions during the storage of diamonds in the mantle or short mantle residence time before the eruption. In contrast, high-temperature annealing results in the formation of aggregated A centres. Some crystals show a concentration of C centres and their nitrogen aggregation state is higher in the core than that at the periphery. Vasiliev et al. (2020) has recently explained this contradiction as syn-growth incorporation of nitrogen directly as paired atoms (A centre) as it has previously been shown to be the case for synthetic crystals (Palyanov et al. 1997; Yelissyeiev et al. 1996).

Recently it has been found that in natural diamonds from eclogitic xenoliths the detection of paramagnetic OK1 and N3 centres correlated with the abundance of titanium compounds (Nadolinny et al. 2009b, 2012 and 2015). The samples with observed OK1 and N3 centres have low total nitrogen concentration of about 10 ppm because the titanium is the nitrogen getter (Nadolinny et al. 2012). The OK1 centres have only been observed in diamonds of cubic habits (Nadolinny et al. 2012). Furthermore, N3 centres were observed in diamond crystals synthesized in systems with a high titanium content (Nadolinny et al. 2009a). Experiments with HTHP annealing at 7 GPa and 2470 K have demonstrated that in some crystals the EPR spectrum of the N3 centre eventually disappeared and the EPR signal from the OK1 centre appeared (Nadolinny et al. 2015). The optical counterpart of the OK1 and N3 centres are the S1 centre (ZPLs at 503.4 and 510.7 nm) and the 440.3 nm system, respectively (Zaitsev 2001; Nadolinny et al. 2009a, b, 2012). In the diamonds studied the paramagnetic N3 centre and PL with ZPL at 440.3 nm were not observed.

Comprehensive investigations using EPR and photoluminescence methods suggest that the N3 and OK1 centres can be modelled as a Ti atom in the substitutional position and a Ti atom in the structure of a double semi-vacancy with one neighbouring nitrogen atom, respectively (Nadolinny et al. 2009b, 2012, 2015). Previously, density functional theory (DFT) calculations for OK1 and N3 centres did not support models containing both oxygen and titanium impurities (Etymimi et al. 2010). However, a recent hybrid DFT analysis (Czelej et al. 2018) provided strong evidence that the neutral Ti–N and TiV–N complexes are indeed the experimentally observed N3 (titanium–nitrogen) and OK1 (titanium–vacancy–nitrogen) colour centres. It is well known that titanium is a nitrogen getter. Because of crystal growth in a titanium-containing medium, the titanium atoms can fit into the diamond structure as TiN or TiCN molecular units. Internal structure strains may be substantially reduced when large titanium atoms are forced out from their substitutional sites into the double semi-vacancy position (an OK1 defect according to Nadolinny et al. 2009b, 2012).

It has been shown that cubic diamonds have a fibrous internal structure (Moore and Lang 1972; Lang 1974; Orlov et al. 1982; Sunagawa 1990; Ragozin et al. 2017). Cuboid diamonds with specific fibrous internal structures could have crystallized under high supersaturation (Sunagawa 1990). Growth under high supersaturation can induce plastic auto-deformation (Punin 1981). The plastic deformation of the diamond samples in this work is revealed by the features of the internal structure (Fig. 1). These structures appear as a ‘tatami’ pattern (two crossed directions of strain lamination) in the birefringence patterns and electron backscatter diffraction (EBSD) images (Ragozin et al. 2017). The ‘amber’ centres observed in FTIR spectra (see Fig. 6) are also related to plastic deformation (Massi et al. 2005). Besides amber centres, Type Ib diamonds exhibit the nitrogen-vacancy defects (NV"
NV) observed with PL (Hainschwang et al. 2013, Smit et al. 2018). The characteristic luminescence centre with ZPL at 635.7 nm was initially attributed to a positively charged vacancy (Charles et al. 2003) but further experimental study has not confirmed this attribution (Steeds et al. 2000). An EPR study of a series of natural cuboid diamonds showed that there could be both a titanium atom and a vacancy in the structure of defect responsible for the optical system at 635.7 nm (Nadolinny et al. 2015). The diamonds studied are also characterized by additional luminescence centres: N3, H3, S1, NV\textsuperscript{0} and NV\textsuperscript{−} (Zaitsev 2001). This set of defects in diamonds has been suggested to form at the stage of post-growth deformation (Titkov et al. 2015b).

The current understanding of the NV centres has recently been discussed in reviews by Doherty et al. (2013) and Pezzagna et al. (2011). The NV centres are produced through the interaction of substituting N\textsubscript{1} and vacancies that are introduced through either irradiation or deformation (Collins 1982). The absence of radiation damage (green or brown spots) and induced GR1 centres (V\textsuperscript{0}) in the studied diamonds (Nasdala et al. 2013) support deformation as the origin of the vacancies. Plastic deformation forms dislocations in the lattice that may generate vacancies (Hull and Bacon 1984). In the PL spectra of natural untreated diamonds, the intensity of the 638 nm center is usually less than that of the 575 nm centre (Dobrinets et al. 2013). In contrast, the 638 nm centre (NV\textsuperscript{−}) is stronger than the 575 nm (NV\textsuperscript{0}) in PL spectra of the diamonds in this study (Fig. 8). A possible reason for this is that nitrogen C centres serve as donors. NV centres capture the extra electrons and become NV\textsuperscript{−} centres while C centres transform to N\textsuperscript{−} (X centres). NV defects have rather low-temperature stability and may anneal out completely at temperatures above 1500 °C (Dobrinets et al. 2013).

5. Conclusions

FTIR spectroscopy data indicate that in yellow cuboid diamonds from the north-eastern Siberian platform the nitrogen is predominantly presented in C and A forms. The lack of nitrogen in B form testifies to the low temperature or short mantle residence time of the diamond genesis. The observed higher aggregation in the peripheral zones of some studied diamonds might be explained by syn-growth incorporation of nitrogen directly as paired atoms. EPR observation has revealed Ti-related OK1 defect in yellow cuboids which could result from the reduction of strains induced by plastic deformation. The formation of NV centres can also be explained by plastic deformation. These new spectroscopic data suggest a plastic deformation within yellow cuboid diamonds which could occur synchronously with their growth. Such cuboid diamonds are suggested to grow fast under high supersaturation. This follows from the manifestation of specific fibrous internal structures that can cause plastic auto-deformation.

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