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Analysis of type IIb synthetic diamond using FTIR spectrometry

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Abstract. Analysis of internal structure in large IIb-type high pressure-high temperature (HPHT) synthetic single-crystal diamond are presented. The concentration of boron impurity in different growth sectors varies from 0.02 to 10.3 ppm. It is shown that in the manufacturing of synthetic diamond plates, internal inhomogeneities of the diamond should be taken into account; plates with different characteristics can be cut from one diamond, each of which can be used for its own purpose.

1. Introduction

Nowadays, methods of synthesis of single-crystal diamonds came to a high level, thus diamonds of different physical types are grown commercially in laboratories. Diamond plates have extreme hardness and high thermal conductivity, that is why they are in demand for medicine, military and space industries, science applications. They are used as UV and IR windows, components of laser optics, lenses for gyrotrons, monochromators and X-ray equipment, sensors and detectors of particles. There are two main competing with each other methods of diamond growth: high temperature – high pressure (HPHT) and chemical vapor deposition (CVD). In this paper, we present data on internal inhomogeneities of 10.87 ct IIb type synthetic single-crystal diamond grown by high pressure-high temperature method.

Synthetic HPHT single-crystal diamonds have sectorial structure (figure 1c). Presence and predominance of different crystallographic forms in synthetic diamonds depends on metals catalysts and an arrangement of seeds in a growth cell (figure 1 a,b). Using of an alloy catalyst based on Fe with incorporation of Aluminum is the cause of {111} and {100} facets predominance, while incorporation of Co, Ti, Sr leads to {113}, {110} and {115} facets development [2].

2. Materials and methods

11.46x11.04x0.51 mm single-crystal diamond plate was cut from a synthetic diamond of IIb type (crystal weight is 10.87 ct) perpendicularly to [111]. This diamond was grown on (111) seed. Except predominant {111} facets, there were different facets of {100}, {110}, {113} and {115} orientations in the diamond. This crystal was dark blue due to strong doping of boron to initial fusion mixture. Studied single-crystal diamond plate had blue color zonality (figure 2) clearly visible by naked eye.

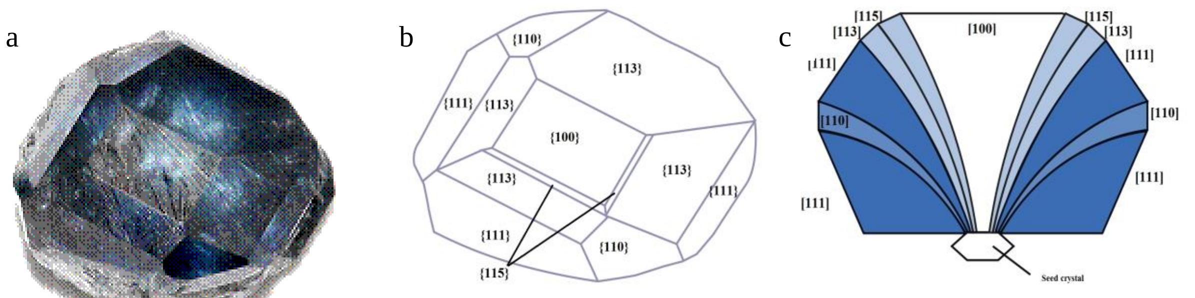

Figure 1. Synthetic HPHT 17.50 ct diamond from "New Diamond Technology" company: a-photo of the crystal, b-scheme of dominant facets, c-scheme of growth sectors.

Figure 2. The plate from the synthetic single-crystal 10.87 ct diamond of IIb type.1, 2, 2a, 3, 4, 5 – IR spectrum points.

FTIR spectra were recorded on Vertex 70 spectrometer with Hyperion1000 microscope with the resolution of 4 cm^{-1} (32 scans averaging). FTIR spectra were registered in 6 points corresponding to main growth sectors with different blue color saturation. Presence of boron in the diamond was determined by FTIR spectra by a number of bands: 1290, 2460, 2802, 2936 cm^{-1} [3]. Generally, concentration of boron was calculated by 2802 cm^{-1} band (figure 4), but if the concentration of boron was extremely high, then it was calculated by 1290 cm^{-1} band (figure 3), because it is impossible to find distinct peaks in three-phonon area in such situation due to strong absorption of boron.

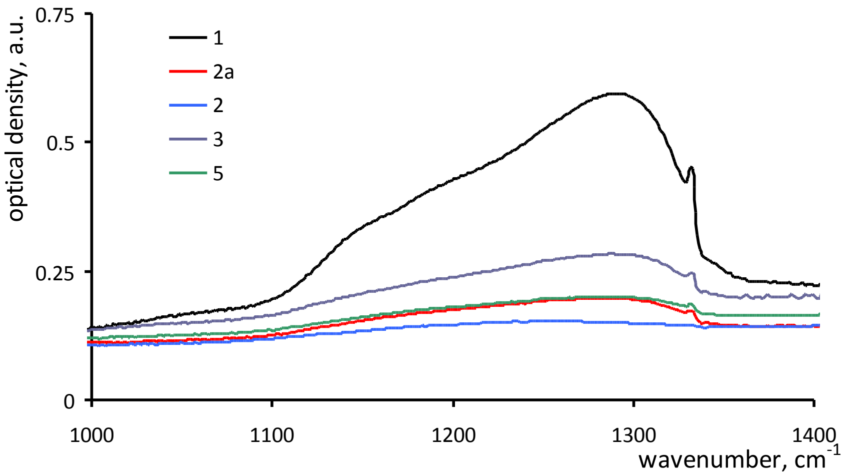

Figure 3. FTIR spectra for tested points (number refers to figure 1) in the range 1000-1400 cm^{-1}.

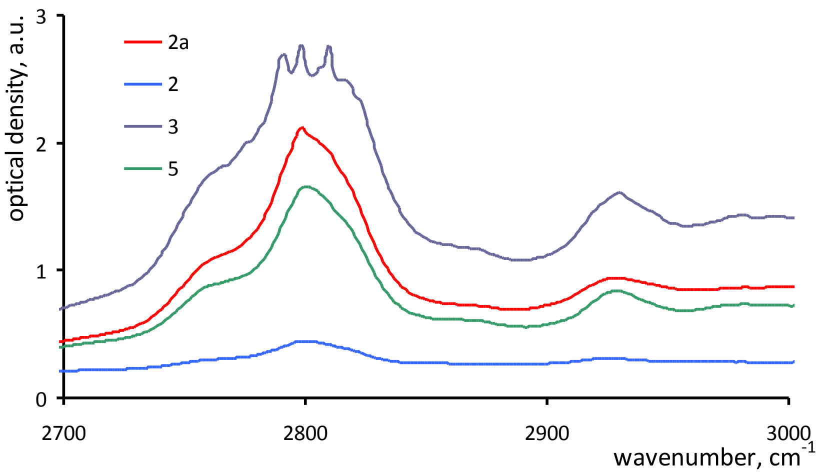

Figure 4. FTIR spectra for points 2, 2a, 5 in the range 2700-3000 cm^{-1}.

3. Results and discussion

Calculations were made by the following equations:

$$[B](ppm)=(5.53\times10^{-4})\times I_{2802}(cm^{-2}) \quad (1)$$

$$[B](cm^{-1})=(2.1\times10^{17})\times \alpha_{1290}(cm^{-1}) \quad (2)$$

where α = A/ t – is an absorption coefficient (A – optical density, t – sheet gage); I – is integral absorbance of peak [1,3-6].

Values of boron concentration from 0.02 to 10.29 ppm (table 1) were obtained. They show that concentrations of boron impurity in a diamond can be varied by 500 times. The most saturated was [111] sector (points 1 and 4). By contrast, the minimum value was registered in [100] sector. Inconspicuous change of intensity of light blue color within light zones of the plate was observed. In fact the value of boron concentrations was 50 times different (points 2 and 2a). This inconspicuous color zonality was explained by the fact that except[100] sector, there are other growth sectors in the diamond, e.g. [110], [113] and [115], which have their own growth nature and are characterized by low concentrations of boron impurity. For example, the point 3 corresponds to the growth sector [110], and here the value of boron concentration is higher, than in sector [100]. In the exit zone to the lateral surface of {113} and {115} facets, the IR spectrum of a point 5 was made.

Table. 1. Calculated concentration of boron for 6 points of a plate.

Point No	Concentration of boron (ppm)	Growth sector
1	10.29	[111]
2a	0.99	[113], [115]
2	0.02	[100]
3	1.95	[110]
4	9.36	[111]
5	0.79	[113], [115]

We can produce monosectorial homogeneous plates of different orientations 5x2, 3x3, 2x2 and 4x3 mm size from the investigated plate. Plates with the concentration edges of boron impurity (from ppb to ppm) can be prodused. Generally, we can produce square monosectorial plates 2x2, 3x3, 4x4, 5x5 mm size and different thickness (0.1-3 mm) with the concentration of boron from level ppb to 10-15 ppm. The extended monosectorial plates can reach 7x5, 9x7 in size. Multisectorial square plates [100] and [111] with boron are able to reach sizes 8x8 up to 10x10 mm.

It should be noticed that inclusions of metals carbides [2] in different growth sectors also create inhomogeneity in boron impurity distribution –white areas with lower concentration of boron around inclusions were observed.

4. Conclusions

- IIb synthetic HPHT diamonds have strong inhomogeneity of boron impurity distribution: concentration in different growth sectors can be 500 times different.
- The sector[111] is the most active for boron impurities acceptance, the [100] sector - the least; [110], [113] and [115] sectors also participate in creation of the composite picture of zonality and are characterized by low values of boron concentration.
- Inclusions of metals carbides in synthetic diamonds also bring inhomogeneity in distribution of boron impurity on a crystal/plate;
- We can produce monosectorial and multisectorial plates of different sizes (up to 10x10 mm) and of different orientations ([111],[100],[110],[113] and [115]) with the concentration of boron from ppb to ppm level.

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