

# SRT research of the crystal defects on the TGT synthesized diamond doped with boron and phosphorus

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**Abstract.** Crystal defects of two synthesized diamonds doped with boron and phosphorus are compared by synchrotron radiation topography (SRT). The crystal defects in the specimen are mainly dislocations. The dislocations are assembled in bundles in the cone-shape and distributed in the directions approximately towards to  $\langle 112 \rangle$  and  $\langle 111 \rangle$ . The Burgers vectors of most of the dislocations are parallel to  $[202]$  and oblique to the dislocations, showing that the dislocations are of mixed type. The features of the crystal defects are quite different in several aspects compared with the diamonds without phosphorous doping. The ionic radius of phosphorous is 118.75 percent larger than that of carbon and intensive distortion may occur near the phosphorous ions. The dislocations thus originated in bundles to release the stress caused by the phosphorous doping.

## 1. Introduction

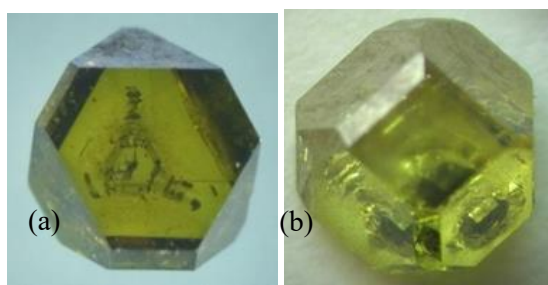
Diamond is widely used in various areas, such as mechanics, thermotics, electrics, magnetics and optics as a result of the excellent characters it is possessed. Since semiconductor component made from diamond can be used at high temperature, high pressure and strong radiation conditions, its application is wide and fascinating in the semiconductor and microelectronics areas [1-3]. The techniques of the P-type doped diamond are well developed nowadays which are mainly doped with boron but the N-type doped diamond are more difficult so its application is restricted [4, 5]. The atoms of the N-type doped diamond are lithium, sodium, nitrogen, phosphorus, oxygen and sulfur, etc.[6, 7]. Phosphorous is the atom mostly be researched except nitrogen in the N-type diamond. Diamond film doped with phosphorous is widely be considered nowadays but the diamond crystal doped with phosphorous is rarely be concerned, especially the crystals synthesized by HPHT techniques [8, 9]. The diamond crystals synthesized by HPHT techniques doped with phosphorous and boron is studied by SRT in this paper. X-ray topography is a convenient method used to reveal the features of the crystal defects, although some other methods have also been used [10-13]. The features of the defects are revealed with the synchrotron radiation topography and compared with that in the diamonds without phosphorous doping. It is concluded that the unique feature in the specimen tested in the experiments maybe resulted from the doping of phosphorous.



## 2. Experiments

### 2.1. Specimens

The specimens D5 and D3, as shown in figure 1 (a) and (b), were synthesized in the State Key Laboratory for Superhard Materials, Jilin University by HPHT temperature gradient growth method. The crystal D5 is a octahedron combined with cube and small faces of rhombododecahedron. Specimen D3 is a cube combined with octahedron and small trapezohedron and rhombododecahedron. The crystal D5 is tawny in color, 3.96mm×3.72mm×1.64mm in dimension and 0.058gram in weight. The specimen D3 is yellowish green, weighted 0.052 gram and 3.84mm×3.25mm×1.72mm in dimension.



**Figure 1.** The diamond crystals D5 and D3. D5 is tawny in color, 3.96mm × 3.72mm × 1.64 mm in dimension and 0.058 gram in weight. D3 is yellowish green, 3.84mm × 3.25mm × 1.72mm and weighted 0.052gram.

### 2.2. Infra-red spectrum experiments

A Tensor 27 infrared spectrometer was used to complete the experiments at The College of Materials Science and Engineering, Yanshan University. The resolution ratio is less than 0.5 cm<sup>-1</sup>, wave-number ranges from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. Compressed potassium bromide tablet is used as the background. Thirty two times had been scanned for both back ground and the specimen. The equipment was preheated for thirty minutes before the experiments.

### 2.3. Synchrotron radiation topography experiments

The synchrotron radiation topography experiments were completed at Beijing Synchrotron Radiation Facility (BSRF). The length of beam 4W1 we used was 43m and the maximum receipt angles were 1.0mrad and 0.3mrad in horizon and vertical, respectively. The energy and intensity of the beam were 2.2GeV and 59.6mA. The Laue transmission setting was employed in the experiments. The distance between the specimen and the film was in the range of 25mm and 35mm. The exposure time was 0.5 to 1 seconds. The FUJI50 film was used. In order to avoid ghost images the back surfaces of the films were not developed.

A series of topographs were taken with different incident directions of the synchrotron radiation beam. The specimens were installed on a triaxiality mount. A photograph was taken when [111] of the specimens was parallel to the incident beam and [11 $\bar{2}$ ] downward. Photographs were then taken after the specimens were turned 15, 30 and 45 degrees clockwise and anticlockwise around the vertical axis.

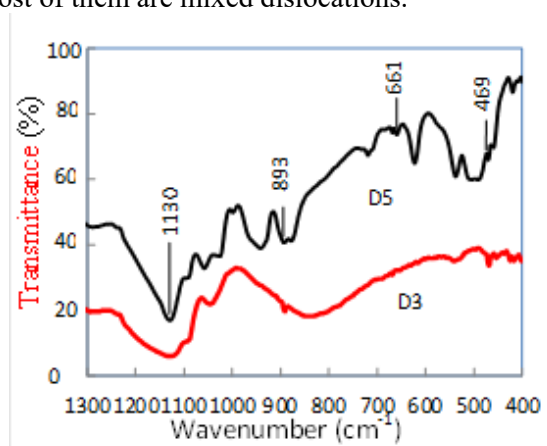
## 3. Results and discussions

### 3.1. The infra-red spectrum experiments

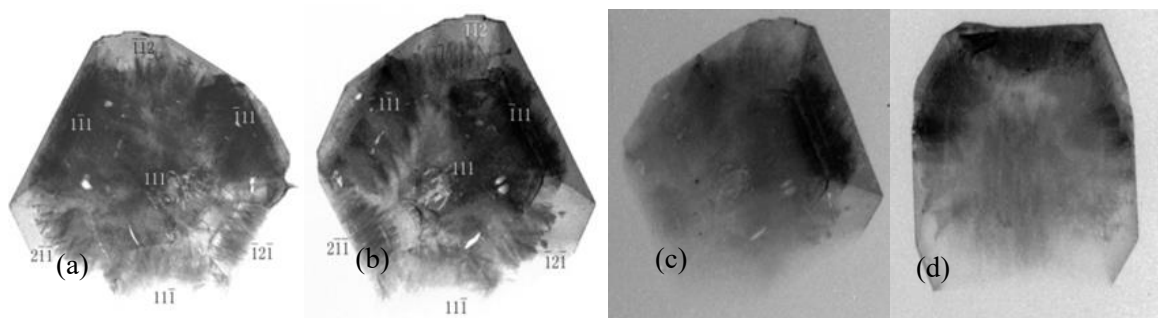
The infrared spectrum diagrams are shown in figure 2. The diamond specimen D3 and D5 belongs to Ib type since absorption bands near 1130 cm<sup>-1</sup> can be seen on both curves of them [14]. Weak absorptions at 469 cm<sup>-1</sup> and 893 cm<sup>-1</sup> in D5 are resulted from the doped boron. The absorption at 661cm<sup>-1</sup> is induced by the impurities of both boron and phosphorous doped within the diamond. The IR result shows that the diamond D5 is doped with both boron and phosphorous. Compared with D5, the absorption at 661cm<sup>-1</sup> in curve of D3 is disappeared. It can be concluded that the specimen D3 was doped with boron but without phosphorous doping [15].

### 3.2. The synchrotron radiation topography experiments

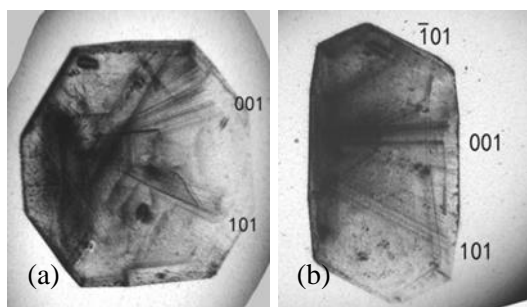
The synchrotron radiation topographic photos of specimen D5 are shown in figures 3 and 4. The crystal defects in the diamond are mainly dislocations and most of them are distributed in  $\langle 112 \rangle$  and  $\langle 111 \rangle$  growing directions. In each area the dislocations are gathered in several bundles and each bundle is composed by dozens of dislocations. The dislocations in a bundle are generated from a same point and extended to cone shaped surface. The vertex of the cone is towards the inner of the crystal and the bottom towards the surface. The angles of the cones are between 20 to 40 degrees. The lengths of the dislocations are between 0.46mm to 0.57mm, average at about 0.5mm. The images of these dislocations are weakened or nearly disappeared in photos (c) and (d) in figure 3, so the product of the diffracted vectors of (c) and (d) in figure 3,  $\vec{g}_{\bar{1}11} \times \vec{g}_{\bar{1}\bar{1}1} = [202]$ , can be a probable estimate of the Burgers vectors of most of the dislocations. The angles between the Burgers vector and the dislocations are bevel so most of them are mixed dislocations.



**Figure 2.** The absorption near 1130 cm<sup>-1</sup> indicates that the diamonds D5 and D3 belong to Ib type. Absorptions at 469 cm<sup>-1</sup> and 893 cm<sup>-1</sup> were resulted from the boron doped in both D3 and D5. The absorption at 661cm<sup>-1</sup> was induced by the impurities of both boron and phosphorous in D5



**Figure 3.** Synchrotron radiation photographs of D5. The diffracted vectors of (a) and (b) are  $[\bar{1}\bar{1}1]$  and  $[\bar{1}11]$ . The dislocations are distributed in the areas of  $\langle 112 \rangle$  and  $\langle 111 \rangle$ . The lengths of the dislocations are about 0.5mm. The diffracted vectors of (c) and (d) are  $[\bar{1}11]$  and  $[\bar{1}\bar{1}1]$ . In photos (c) and (d), the images of the dislocations are very weak or nearly vanished.



**Figure 4.** The synchrotron radiation topographic photos of D3 with diffracted vector  $\vec{g}_{3\bar{1}1}$  in (a) and  $\vec{g}_{202}$  in (b). The dislocations are generated from the seed and extended towards to  $\langle 100 \rangle$  and  $\langle 110 \rangle$ . The dislocations are parallel within each bundle.

Compared with D5, the dislocations in D3 are quite different in several aspects. The directions of the dislocations in the diamond D3 are mainly extended to  $\langle 100 \rangle$  and  $\langle 110 \rangle$ , while in the specimen D5 are to  $\langle 112 \rangle$  and  $\langle 111 \rangle$ . The dislocations in the diamond D3 are generated from the surface of the seed, often, all the dislocations are generated from a unique source, while in D5, numerous sources are observed. The dislocations in D3 are parallel within a bundle, while in D5 the dislocations are scattered in a cone-shaped bundle in the range about 40 degrees.

The differences are resulted probably from the phosphorous doping since the two specimens are synthesized at similar conditions except the phosphorous doping. It could be imagined that the vertex of the cone-shaped dislocation bundle might be the site of the phosphorous ion be doped. The ionic radius of phosphorous and carbon are 0.035nm and 0.016nm, respectively [16]. The ionic radius of phosphorous is about 118.75 percent larger than that of carbon. The crystal lattice near the phosphorous ion doped in the diamond must be strongly distorted. The dislocations bundles are thus generated so that the distortion could be released.

#### 4. Conclusions

Diamond specimens D5 and D3 synthesized by HPHT method with and without phosphorous doping are analyzed with IR and SRT in this paper. The IR experiments show that the specimen D5 is doped with boron and phosphorous while D3 is only boron doped. The crystal defects are revealed by the SRT experiment. In specimen D5, the defects are mainly dislocations which are bundled in cone-shape and situated in the areas of  $\langle 112 \rangle$  and  $\langle 111 \rangle$ . The Burgers vectors of most dislocations are parallel to  $[202]$ , bevel to almost all the dislocations, reveal that the dislocations are of mixed types. Compared with D5, the features of the dislocations in D3 are quite different in several aspects. In D3 almost all dislocations are generated from the seed and grown to  $\langle 100 \rangle$  and  $\langle 110 \rangle$ . The phosphorous doping is the most probably reason that resulted all the differences mentioned above. The ionic radius of phosphorous is about 118.75 percent larger than that of carbon, so the crystal lattice is strongly distorted near the phosphorous ions. The dislocations are thus generated around the doped phosphorous ions in order to release the stress brought from the lattice distortion.

#### Acknowledgment

The SRT experiments were completed at BSRF. The diamond specimens were synthesized by the National Laboratory, Superhard Materials, Jilin University.

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