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HPHT synthesis and crystalline quality of large high-quality (001) and (111) diamond crystals

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ABSTRACT

High-quality type IIa diamond crystals measuring up to 12 mm in diameter (8 to 10 carats) were successfully grown by the temperature gradient method at high pressure and high temperature (HPHT) on defect-free (001)-oriented and (111)-oriented seed crystals. The crystalline quality of the grown crystals was qualitatively evaluated by synchrotron based X-ray topograph and rocking curve (RC) measurements. The results revealed that the crystals grown on (001)-oriented seeds have extremely high quality in the (001) growth sector, which extends straight upward from the seed surface. This sector contains very few dislocations and stacking faults in the upper part in particular, where the RC almost agrees with the theoretical curve. For the crystals grown on (111)-oriented seeds, it was confirmed that crystal defects are fewer in the outer (100), (010), and (001) growth sectors than in the central (111) growth sector extending straight upward from the seed surface. The RCs in selected regions of these {100} growth sectors are close to the theoretical curve, indicating high crystalline quality.

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1. Introduction

Diamond crystals synthesized by the temperature gradient method at high pressure and high temperature (HPHT) may be far superior in crystalline quality and consistency among individual crystals compared to natural diamonds. In particular, type IIa diamond crystals synthesized by advanced control of impurities and crystal defects have extremely high crystalline quality [1–4]. They are, therefore, very useful as monochromators or other optical elements of synchrotron X-ray radiation beams [5–10]. The next-generation of highly brilliant beams (free-electron X-ray laser, XFEL) requires diamonds of much higher crystalline quality, and so there are high expectations for these large synthetic type IIa diamond crystals. They are also expected to be introduced to other applications such as various high-sensitivity detectors (sensors), or as substrates for electronic devices in the future.

In the 1990s we demonstrated that high-quality type IIa diamond crystals of 5–6 mm (1–2 carats) in diameter could be produced by eliminating chemical impurities below 0.1 ppm [1] using low-defect density (001)-oriented seeds [2]. Recently, we succeeded in producing much larger high-quality type IIa diamond crystals measuring up to 12 mm in diameter by improving the synthesis technology. The (001) growth sectors of the large crystals grown on (001)-oriented seeds were found to be almost defect-free [11]. In addition, we successfully synthesized large type IIa crystals of 12 mm in diameter with (111)-oriented seeds. In this study, we investigate the distribution of dislocations and stacking faults in these large crystals grown on (001)-oriented and (111)-oriented seeds and the dependency of crystalline quality on the major grown sectors through X-ray topograph and rocking curve (RC) measurement using synchrotron X-ray beams. We also indicate the differences between the crystals grown from these two different orientations of seeds.

2. Experimental methods

High-quality type IIa diamond crystals measuring up to 12 mm in diameter (8 to 10 carats) were synthesized by the temperature gradient method under HPHT conditions of 5.5 GPa and 1340–1350 °C using Ti-loaded high-purity Fe–Co solvents, high-purity carbon sources, and defect-free seed crystals [1,2]. Seed crystals measuring 0.5 × 0.5 mm cut from high crystalline quality type IIa crystals synthesized by the temperature gradient method were used. Two types of seed crystal, (001)-oriented and (111)-oriented, were prepared. The diamond crystals grown on the (001)-oriented and (111)-oriented seed surfaces were sliced by laser cutting as shown in Fig. 1 to prepare diamond plates of 0.8 to 1.2 mm in thickness. The (001) surfaces were polished using metal-bonded diamond wheel. The (111) surfaces were not polished in this study because the (111) plane is too hard and mechanical damage is easily introduced while polishing. UV-excited luminescence images of these diamond plates were taken using the DiamondView™ instrument (Diamond Trading Company, [12]) in order to distinguish growth sectors in the diamond crystals.

The crystalline quality of these diamond plates was qualitatively evaluated by synchrotron X-ray RC measurement and topography as

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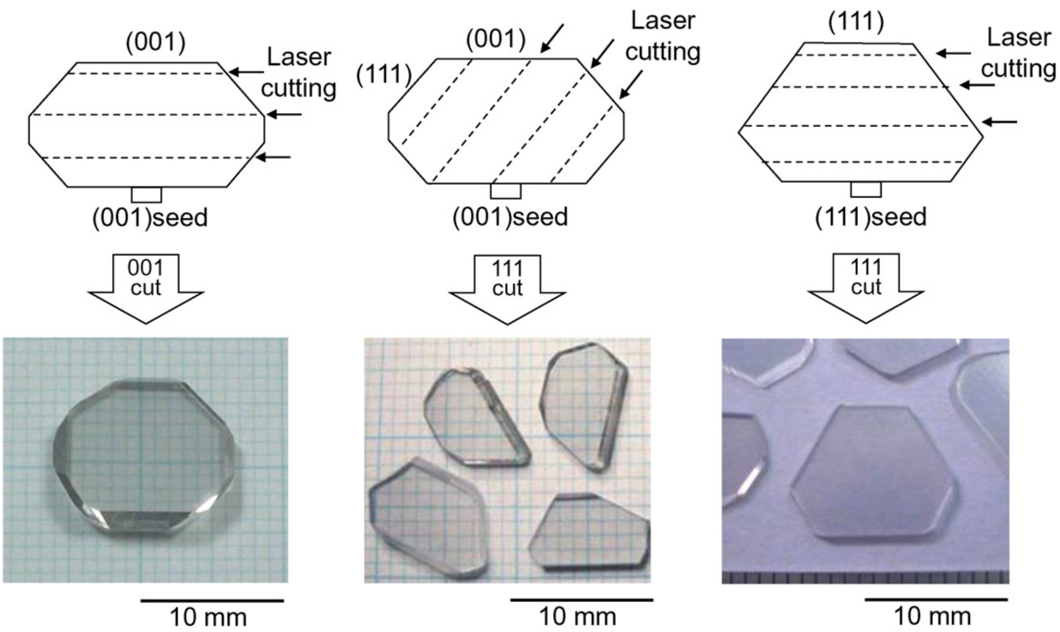

(a) (001)/(001) crystal (b) (111)/(001) crystal (c) (111)/(111) crystal

Fig. 1. Large (001) and (111) high-quality type IIa diamond crystal plates. (a) (001)/(001) crystal: (001) plate cut from the crystal grown on the (001)-oriented seed, (b) (111)/(001) crystal: (111) plate cut from crystal grown on (001)-oriented seed, (c) (111)/(111) crystal: (111) plate cut from crystal grown on (111)-oriented seed.

described next. At the 1-km beamline of SPring-8 (BL29XUL) [13], the topographs were acquired under quasi-plane wave incidence using a Si collimator [14]. Table 1 shows the experimental conditions (reflection plane, collimator, photon energy). A CCD-based X-ray camera with a pixel size of 12 μm was used to capture the topographs. The topographs were acquired at the peak and low-angle side tail region of the RC. The RCs were measured with footprints over the entire surface of the crystals and in a narrow area (250 × 250 μm) selected by a slit.

3. Results and discussion

3.1. Synthesis of high-quality large type IIa diamond crystals

Crystal growth experiments were performed for more than 200 h under very precise temperature control within a narrow high-pressure and high-temperature region where good-quality crystals can be obtained [4,11]. Examples of the resulting large good-quality crystals are shown in Fig. 1. Large diamond plates of both (001) and (111) orientations measuring up to 12 mm in diameter were produced. Fig. 1(a) shows a (001) crystal plate cut out from the central region of the crystal grown on (001)-oriented seed by cutting in the horizontal direction, parallel to the (001) surface. Fig. 1(b) shows a (111) crystal plate cut in an oblique direction, parallel to the oblique (111) surface from the crystal grown on (001)-oriented seed. Fig. 1(c) shows a (111) crystal plate prepared from the crystal grown on (111)-oriented seed by cutting in the horizontal direction, parallel to the (111) seed surface. Here, the diamond crystal plates cut as shown in Fig. 1(a), (b), and (c) are referred to as "(001)/(001) crystal", "(111)/(001) crystal", and "(111)/(111) crystal", respectively. The amount of chemical impurities such as nitrogen and boron in each of these large crystals was less than 0.1 ppm (below the limit of analysis by optical absorption).

Table 1
Experiment conditions.

Reflection plane of diamond	Collimator (asymmetry)	Photon energy (keV)
400 Bragg geometry	Si(531)	19.744
111 Bragg geometry	Si(220)	9.439
220 Laue geometry	Si(331)	14.547

The UV-excited luminescence images of the (001)/(001) crystal plate and the (111)/(001) crystal plate captured with the DiamondView™ instrument are shown in Fig. 2. The {100} growth

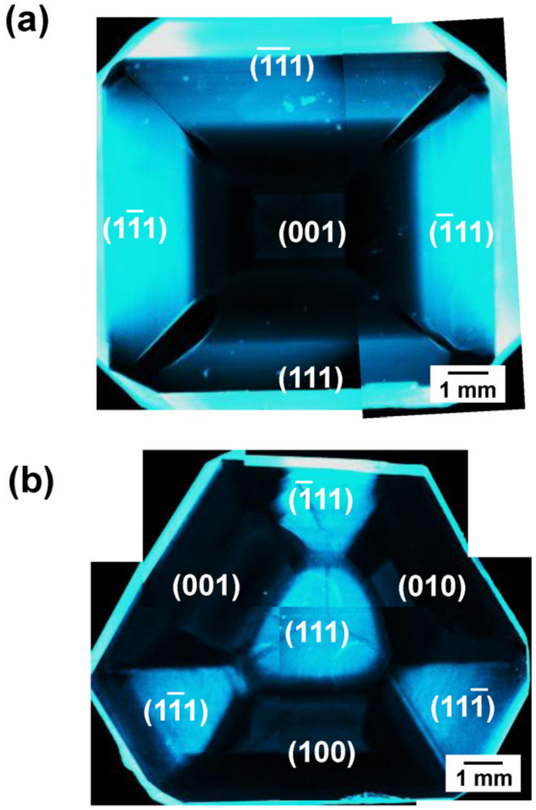

Fig. 2. UV-excited luminescence images. (a) (001) crystal plate cut out from the diamond crystal grown on a (001)-oriented seed, (b) (111) crystal plate cut out from the diamond crystal grown on a (111)-oriented seed. Several images were taken and patched because the diamond crystal plates were larger than the field of view.

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