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Chemical Vapor Deposition of ¹²C Isotopically Enriched Polycrystalline Diamond

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Received April 1, 2012; accepted May 17, 2012; published online August 29, 2012

¹²C isotopically enriched polycrystalline diamond free-standing plates were synthesized by microwave plasma-assisted chemical vapor deposition. Diamond plates 30 mm in diameter and 100–600 μm in thickness were successfully obtained at a typical growth rate of 3-5 μm/h. The ¹²C isotopic enrichment was 99.997%, whereas the nitrogen content was as low as 4 ppb. The preferential orientation of the (110) crystallographic direction normal to the substrate plane was proved using the X-ray diffraction pattern. The sharp Raman peak with a low background and a clear free-exciton recombination emission in a cathodoluminescence measurement at room temperature indicate that the polycrystalline diamond has a substantially high crystalline quality. © 2012 The Japan Society of Applied Physics

1. Introduction

Natural diamond is composed of carbon atoms whose isotopic ratio of natural abundance is 98.9% for ¹²C and 1.1% for ¹³C.¹⁾ In the case of man-made diamond, carbon isotopic control is possible by changing the carbon isotopic ratio in the source materials. Carbon isotopic control is known to improve the physical properties of diamond markedly. Among the physical properties, thermal conductivity is significantly increased by isotopic enrichment. Anthony et al. reported that the room-temperature values of high-temperature/high-pressure (HPHT) single crystals were increased from 22.3 to $33.2 \,\mathrm{W \, cm^{-1} \, K^{-1}}$ by decreasing the ¹³C content in a diamond lattice from 1.1 to 0.07%.²⁾ The quantum states of electron spin in a color center in diamond are also tuned by controlling the nuclear spin density originating from ¹³C. Mizuochi et al. reported that the spin coherence time of the nitrogen-vacancy (NV^-) center in diamond single crystals was prolonged from 0.6 to 1.8 ms by decreasing the ¹³C concentration from 1.1 to 0.1%³⁾ These facts prove that diamond growth technology accompanied by carbon isotopic control is as important as impurity doping for tuning the electrical conductivity.

Thus, there have been several studies describing the growth of isotopically enriched diamond. Diamond crystals used in most of these studies were grown by an HPHT method,^{1,2,4)} whereas there are only a few reports on the chemical vapor deposition (CVD) method.^{5,6)} In particular, the reports on ¹²C-enriched polycrystalline diamond growth are limited, although the polycrystalline diamond is suitable for increasing the crystal wafer size and therefore technologically important. Kataoka *et al.* grew polycrystalline diamond plates, the room-temperature thermal conductivity of 27 W cm⁻¹ K⁻¹ has been reported.⁷⁾ This value is higher than that of type-IIa diamond single crystals (22 W cm⁻¹ K⁻¹), which are isotopic in natural abundance.

In this study, 5N-grade ${}^{12}C$ isotopic enrichment (~99.999%) was performed for polycrystalline diamond growth. The purpose of this study is to demonstrate the highest ${}^{12}C$ isotopic enrichment of polycrystalline diamond among those reported thus far. At the same time, isotopic

contamination during the gas-solid phase conversion by diamond CVD on a foreign substrate is investigated. Another purpose of this study is to investigate the growth features of polycrystalline diamond under the high-plasmadensity diamond CVD conditions.^{8,9} Impurities, such as nitrogen, and/or defects in the crystal strongly affect the diamond physical properties, such as thermal conductivity.¹⁰ Thus, a high purity and a high crystalline quality as well as the desired film texture are required for attaining the extreme physical properties expected for highly isotopeenriched polycrystalline diamond.

2. Experimental Procedure

Undoped polycrystalline diamond films were deposited by microwave plasma-assisted chemical vapor deposition (MPCVD). A molybdenum circular plate with a diameter of 30 mm and a thickness of 5 mm that has been commercialized as a sputtering target (Furuuchi Chemical) was used as a substrate. The purity of the Mo plate was 99.95%. To suppress unintentional ¹³C contamination, neither the diamond seeding process¹¹⁾ nor the bias enhancement procedure for accelerating diamond nucleation¹²⁾ was applied in the initial stage of diamond growth. The MPCVD system used in this study was designed in the National Institute for Materials Science (NIMS). A schematic design of the early NIMS-type MPCVD system has been reported elsewhere.¹³⁾ The reaction chamber was stainless steel, and both the inner cavity and the sample stage were water-cooled. The top metal plate of the sample stage was made of molybdenum.

The system was equipped with a microwave generator whose maximum power was 3 kW (Nissin MPS-30W), a microwave auto matching controller (Nihon Koshuha AMC-9701), directional couplers (Nissin PM-60) and an isolator (Nissin NISJ-60W) connected to each other with waveguides. A transverse electric (TE) mode microwave emitted by the generator propagated along the waveguides and the above-described components. Finally, the microwave was introduced into the reaction chamber through a waterless quartz window after the microwave mode conversion from the TE mode to the transverse magnetic (TM) mode at a point just above the reaction chamber. The reaction chamber was evacuated by a 400 L/s magnetically suspended turbomolecular pump (TMP; Osaka Vacuum TG420M) to up to <10⁻⁶ Pa before diamond growth. The TMP, the mechanical

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booster pump, and the rotary pump were connected in series. Then, the reaction gas was evacuated through the TMP during the diamond CVD process, which is efficient for minimizing the unintentional incorporation of impurities, such as ¹³C and nitrogen, due to back diffusion from the oilsealed pumping system. The source gas was a mixture of H₂ purified by a palladium purifier (Japan Pionics LS-3) and methane specified to 99.999% ¹²C isotopic enrichment (Cambridge Isotope Laboratories CLM-392). Since the ¹²Cenriched methane contained a certain amount of residual gas such as 14 ppm nitrogen, the methane was purified by a zirconium purifier (SAES Getters PS4-MT3) to obtain a 9Ngrade chemical purity. The gas feeding system used in this study was equipped with several bypass lines of mass-flow controllers to evacuate residual gases, which was effective to suppress the incorporation of the ¹³C mixture into the diamond film when the methane source gas was switched from the natural abundance methane to the ¹²C-enriched one. The diamond growth parameter ranges of MPCVD were as follows: the reaction pressure range of 100-180 Torr, the microwave power range of 1.0-1.7 kW, the methane concentration ratio range of 4-10%, and the substrate temperature range of 900-1050 °C. The typical growth rate was $3-5\,\mu\text{m/h}$ under the high-density plasma condition of ~ 70 W cm⁻³. ¹²C-enriched diamond plates 30 mm in diameter and 100-300 µm in thickness were synthesized.

The diamond free-standing plates were characterized using an optical microscope equipped with Nomarski-mode filters (Olympus BX60), a laser microscope (Keyence VK-9700), and devices for Raman spectroscopy (Photon Design PDPX), X-ray diffraction (XRD; Rigaku RINT-2000), cathodoluminesence (CL) spectroscopy (Topcom SM-350), secondary ion mass spectrometry (SIMS; Cameca Ametek IMS-7f), and electron spin resonance (ESR) spectroscopy (Bruker E500).

3. Results and Discussion

Figure 1(a) shows a photograph of the polycrystalline diamond grown on the entire area of the Mo substrate. The film thickness at the substrate fringe was larger than that at the center part and the thickness variation was about 10%. After the MPCVD growth, the diamond plate was spontaneously removed from the Mo substrate when the diamond was thicker than 200 μ m, attributed to the difference in thermal expansion coefficient. However, this difference sometimes caused the diamond plate to break into several pieces, as shown in Fig. 1(b). The obtained diamond plate was transparent.

Figure 2(a) shows a typical laser microscopy top-view image of the grown polycrystalline diamond plate with a thickness of $\sim 200 \,\mu$ m. The image was taken from the growth face. The laser wavelength for observation was 408 nm and the height accuracy was 14 nm. The grain size was varied in the range of 10–50 μ m and the change in the grain size with the increase in film thickness was small. Figures 2(b) and 2(c) show a bird's-eye-view image of the sample and its line height profile, respectively. The surface roughness R_a was estimated to be $\sim 2 \,\mu$ m.

Figure 3(a) shows the wide-range Raman spectra of three polycrystalline diamond samples grown under different conditions. The spectrum taken from a homoepitaxial diamond



Fig. 1. (Color online) Photograph of polycrystalline diamond grown on the entire area of a Mo substrate: (a) before and (b) after removal.



Fig. 2. (Color online) Typical laser microscopy images of the grown polycrystalline diamond: (a) top view, (b) bird's-eye view, and (c) height profile.



Fig. 3. (Color online) Raman spectra of three polycrystalline diamond samples. The homoepitaxial diamond grown on the IIa (100) diamond single crystal is shown as a reference. (a) Wide-range and (b) magnified spectra around the diamond Raman peak.

 Table I.
 Raman peak position and its FWHM for each sample.

Sample name	Raman peak position (cm^{-1})	FWHM of Raman peak (cm ⁻¹)
Poly A	1332.51	2.72
Poly B	1331.94	4.65
Poly C	1332.85	3.90
Homoepitaxial on IIa (100)	1332.19	2.55

film grown on a type-IIa (100) diamond single crystal was shown as a reference. No remarkable signals reflecting sp^2 phases were detected at ~1600 cm⁻¹, suggesting that the quality of the polycrystalline diamond is high compared with that of the conventional polycrystalline diamond. The spectra magnified at around $1332 \,\mathrm{cm}^{-1}$ are shown in Fig. 3(b) and the peak fitting results obtained using the single-peak Gaussian function are listed in Table I. The diamond Raman peak was shifted to a higher wavenumber by $0.3-0.7 \,\mathrm{cm}^{-1}$ from the position of the single crystal, indicating the presence of compressive strain. By using the reported correlation factor between the wavenumber and the internal stress of $3 \text{ cm}^{-1}/\text{GPa}$,¹⁴⁾ and the wavenumber of the stress-free diamond Raman peak shift, 1332.19 cm⁻¹, obtained from the homoepitaxial diamond film on type IIa (100), the internal stress of the sample Poly A was estimated to be 107 MPa. This value is comparable to the stress near the dislocations in homoepitaxial diamond.¹⁵⁾ Two or three peaks were observed from the sample Poly B. The transparency of Poly B was observed to be the lowest among the diamond plates examined. This indicates that the darker color of the polycrystalline diamond is strongly correlated with the inner strain. The full width at half maximum (FWHM) of the Raman peak of the sample Poly A is comparable to that of the single crystal and the high-quality area of the heteroepitaxial diamond on Ir(100).¹⁶⁾



Fig. 4. (Color online) XRD patterns obtained from the three polycrystalline diamond samples grown under the high plasma density condition and one reference pattern.

Figure 4 shows the XRD patterns obtained from the growth surfaces of these three polycrystalline samples. The XRD pattern of a conventional polycrystalline diamond film deposited on a silicon substrate by the hot filament method was plotted as a reference. Typically, the {220} diffraction intensity is lower than the {111} diffraction intensity for a conventional randomly oriented polycrystalline diamond.¹⁷⁾ In contrast, the {220} peak intensity was found to be higher than the {111} peak intensity for the present samples grown under the high-density plasma condition.

Then, the diffraction intensity ratio of {220} to {111} was plotted as a function of the crystal thickness of polycrystal-



Fig. 5. (Color online) Diffraction intensity ratio of {220} to {111} plotted as a function of the crystal thickness of the polycrystalline diamond. The inset shows the Raman peak width as a function of $\{220\}/\{111\}$ ratio. The inset shows the correlation between the Raman peak width and the XRD {220}/{111} intensity ratio.

line diamond, as shown in Fig. 5. The gray band and the data indicated by an orange rectangle in Fig. 5 are those of the conventional polycrystalline diamond used as the reference. It is clearly seen that the intensity ratio $\{220\}/\{111\}$ increases with the growth thickness. This indicates that the sample has a preferential crystalline orientation in the (110)direction normal to the substrate plane. The preferential orientation of grains in polycrystalline diamond has been reported by several research groups.¹⁸⁻²⁰⁾ A model describing the (110) preferential orientation was proposed by Drift;²¹⁾ the orientation is a consequence of the growth competition between different diamond grains. In other words, after the random nucleation of diamond crystals in the initial growth stage, the crystals with the highest growth rate in the direction of the surface normal continue to grow at the expense of less favorably oriented ones. In this manner, a limited number of crystallites survive after a longer growth duration, resulting in the formation of the highly textured film consisting of larger, columnar crystallites.

The inset in Fig. 5 shows the correlation between the FWHM of the Raman peak and the $\{220\}/\{111\}$ intensity ratio of the XRD pattern. It clearly shows that the FWHM decreases with increasing degree of the $\langle 110 \rangle$ preferential orientation of the polycrystalline diamond surface. This indicates that the polycrystalline diamond quality can be deduced from the $\{220\}/\{111\}$ intensity ratio.

To investigate the preferential orientation, a diamond plate with a thickness of $610 \,\mu\text{m}$ was cleaved in two near the center of the plate, and then the cleaved face was observed using the optical microscope. Figure 6(a) shows the cross section of the plate, and the magnified image taken in the region indicated by a yellow rectangle is shown in Fig. 6(b). Here, the bottom is the nucleation side, whereas the top is the growth side. The results indicate that the grains extended in the growth direction for more than 200 μ m while keeping a column width of ~50 μ m.

Figure 7 shows the typical CL spectra taken from the growth surface of the sample Poly A. As shown in Fig. 7(a),



Fig. 6. (Color online) Optical microscopy image of the cross section of the plate: (a) Low magnification image and (b) magnified images of the region in (a) indicated by a yellow rectangle.

the free-exciton recombination (FE) emission at 235 nm and the band-A emission at ~430 nm attributed to dislocations are observed at room temperature. Although the FE intensity is lower than that from the homoepitaxial diamond (100) film,²²⁾ the clear FE emission indicates that the crystalline quality of the polycrystalline diamond is high, consistent with the results of Raman spectroscopy. Figure 7(b) shows the spectrum taken at 80 K. The free-exciton recombination emission relative to the band-A emission increases with decreasing measurement temperature.

Figure 8 shows the CL image taken at the wavelength corresponding to the (a) free-exciton recombination and (b) band-A emissions. Arrows with the same color in each figure indicate the same position of the grains in the sample. Dark parts were observed in the band-A image, whereas the corresponding FE image was complementally bright,²³⁾ indicating that the interior portion of the grains has a high crystalline quality with a low dislocation density.

Finally, the isotopic and chemical purity of the grown diamond was evaluated by SIMS and ESR measurements. SIMS was carried out for the polycrystalline diamond to evaluate the isotopic enrichment. Cs^+ accelerated to 15 kV was used as the primary ion for the evaluation of the isotopic enrichment, whereas O_2^+ accelerated to 10 kV was applied to the highly sensitive detection of unintentionally incorporated boron in diamond. Figure 9 shows the depth profiles of the ¹²C and ¹³C secondary ion intensities up to $2.5 \,\mu\text{m}$ in depth from the grown surface. It also shows that the ¹²C concentration is 99.997%. Thus far, this isotopic enrichment is the highest among the reports on the ¹²C enrichment of diamond by methods including both the HPHT and CVD methods. The high concentration of ¹³C near the surface may



Fig. 7. (Color online) Typical CL spectra of the sample Poly A taken at (a) 300 K and (b) 80 K.



Fig. 8. (Color online) CL image taken at the wavelength corresponding to the (a) free-exciton (FE) recombination and (b) band-A emissions.

come from the effect of an adsorbed carbon impurity. Since the value is almost comparable to the isotopic enrichment of the source methane gas of 99.999%, the unintentional ¹³C incorporation was suppressed effectively in the growth



Fig. 9. (Color online) SIMS depth profiles of ${}^{12}C$ and ${}^{13}C$ of the polycrystalline diamond plate.

system employed in this study. The nitrogen, boron, and hydrogen concentrations were below the detection limits of the SIMS measurements of 2×10^{16} , 1×10^{14} , and 1×10^{14} cm⁻³, respectively.

The nitrogen density was also estimated from the ESR measurements of the P1 center arising from the isolated substitutional nitrogen in the neutral charge state (S = 1/2).²⁴⁾ In the undoped CVD diamond, the P1 center is the dominant configuration of the nitrogen impurity. The rapid passage technique with a high sensitivity for determining P1 concentrations of 0.1 ppb to 1 ppm was employed.²⁵⁾ The P1 center incorporated into the grains is useful as a probe to confirm the (110) preferential orientation. The ESR spectrum of P1 consists of three lines arising from the ¹⁴N (I = 1, natural abundance 99.64%) hyperfine interaction. In the single crystal, the line position of the ESR spectrum of P1 (C_{3v} symmetry) varies with the orientation of the magnetic field (B) with respect to the crystal axes since the ¹⁴N hyperfine interaction is anisotropic ($A_{\parallel} = 4.093 \text{ mT}$, $A_{\perp} = 2.920 \,\mathrm{mT})^{26}$ and there are four sites corresponding to four (111) directions. The central hyperfine line is nearly isotropic with a very small g-anisotropy ($g_{\parallel} = 2.0024$, $g_{\perp} = 2.0025$ ²⁷⁾ A single-crystal ESR spectrum of P1 with $\boldsymbol{B} \parallel [110]$ consists of five lines with the intensity ratio 2:2:4:2:2, since there are two sets of two magnetically equivalent sites in this orientation. In uniaxial (110) growth, the grains (small single crystals) are preferentially aligned with the (110) axis perpendicular to the plate, while the orientation in the plane is random. The ESR spectrum taken with \boldsymbol{B} perpendicular to the plate plane gave five lines with the same positions as those obtained for a single crystal with **B** \parallel [110]. The outer hyperfine lines exhibited broadening caused by orientation disorder, in which the grains are misaligned slightly with respect to each other. The ratio of the signal intensity obtained by integration was 2:2:4:2:2. On the other hand, for B parallel to the plate plane, the ESR spectrum of the P1 of random orientation was obtained. The P1 concentration in the preferentially oriented grains obtained for the sample Poly A was as low as $\sim 4 \text{ ppb}$ $(7 \times 10^{14} \text{ cm}^{-3}).^{24}$ Scanned confocal fluorescence microscopy showed NV centers inside the grains, mostly in single NV centers (i.e., at a concentration lower than 0.1 ppb). The coherence time of ~2 ms obtained for the single NV centers, which was comparable to the best time (1.8 ms) reported for NV in single-crystal CVD films with 99.7% ¹²C enrichment, indicated the high purity and quality (low defect density) of the grains.²⁴⁾ Further investigation, which is technologically important for the practical application of the ¹²C-enriched polycrystalline diamond will be carried out over a larger area.

4. Conclusions

¹²C isotopically enriched polycrystalline diamond freestanding plates were grown by microwave plasma-assisted chemical vapor deposition under a high-density plasma condition. The polycrystalline diamond plates thus grown have a high quality as evidenced by Raman and CL spectroscopy results. XRD revealed that the polycrystalline diamond has a preferential crystalline orientation in the $\langle 110 \rangle$ direction. A ¹²C isotopic enrichment of 99.997% with a low nitrogen content of 4 ppb was confirmed by SIMS. The ¹²C highly enriched and high-quality polycrystalline diamond is expected to open up new fields for diamond research and development.

Acknowledgements

This work was partly supported by a Grant-in-Aid for Scientific Research from the Japan Society for the Promotion of Science (No. 23360143) and the Strategic International Cooperative Program (Nanoelectronics) from the Japan Science and Technology Agency.

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SELECTED TOPICS IN APPLIED PHYSICS



Meiyong Liao received the Bachelor and Master degrees from Lanzou University in 1996 and 1999, respectively, and the Ph. D. degree from the Institute of Semiconductor, Chinese Academy of Sciences, China in 2002. From 2002 to 2004, he was a visiting researcher in Kyoto University. He joined in National Institute for Material Science, Japan, as a postdoctoral researcher from 2004–2008. In April 2008, he was promoted as the senior researcher in NIMS. His current research fields are diamond

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