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X-Ray Topographic Study of a Homoepitaxial Diamond Layer on an Ultraviolet-Irradiated Precision Polished Substrate

Y. Kato*, H. Umezawa and S.I. Shikata
Advanced Industrial Science and Technology (AIST), Ikeda, Osaka 563-8577, Japan

Suitable techniques for the growth of high-quality single-crystal diamond are needed in order to use single-crystal diamond in power devices. Because the ion plantation technique cannot be used for diamond doping, a drift layer and a conduction layer for a diamond power device were grown by chemical vapor deposition. An important challenge in this field is to reduce the dislocation density in the epitaxial layer. The dislocation density was found to increase during the chemical vapor deposition process. Because a defective surface is one cause of increased dislocation density, the use of a UV-polished substrate having no scratches due to mechanical polishing was investigated.

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1. Introduction

Semiconducting diamond has attracted considerable attention for its use as a material for power devices owing to its high breakdown characteristics and high carrier mobility in high-temperature, high-voltage environments [1]. Recently, the development of a diamond Schottky barrier diode that exhibits stable performance at temperatures greater than 200 °C has been reported [2–4]. For the development of high-performance devices, the density of defects in the epitaxial layer is a critical issue [5, 6]. The permissible defect concentration can be determined using Murphy's yield model [7]. For example, using the model, one can obtain the permissible defect density for a high-current device. By setting the electrode size to 1×10^{-4} cm^2 and using Murphy's yield model, while assuming the performance of the electrode deposited on a threading dislocation to be poor, one can find the dislocation density. If the dislocation density is approximately 10^4 cm^{-2}, most of the electrodes (more than 60%) will be inferior. The results of calculations based on Murphy's yield model suggest that high-quality diamond, which has a defect density of less than 10^3 cm^{-2}, is essential for developing devices with sufficiently high performance.

Techniques for growing high-crystalline-quality single-crystal diamond under high-pressure and high-temperature conditions (also known as HPHT diamond) have been developed by several researchers [8, 9]. The defect density of commercially available diamond is approximately 10^3–10^5 cm^{-2} [10]. Diamond-based power devices have a multilayered structure, and HPHT diamond can be used as only the substrate in the multilayered structure. For use in other layers, high-quality doped diamond is required, and it is difficult to produce high-quality doped diamond via ion implantation. For chemical-vapor-deposited diamond (CVD diamond), the dislocation density in a limited area of the diamond was reported to be less than 400 cm^{-2} by Martineau et al. [11]. The density of dislocations in an epitaxial CVD diamond layer depends on the defect distribution and defect density of the diamond substrate.

Some researchers have suggested that pretreating the substrate is effective for growing diamond layers with a lower defect density [12, 13]. For example, Mokuno et al. used a flat surface with an average roughness of less than 1 nm [12].

In this study, we investigated the effect of an ultraflat polished substrate on the quality of epitaxially grown CVD diamond by examining X-ray topography (XRT) images. A scaife-polished sample was used as a reference to compare the effects of high-quality polishing. For simplicity, a type-Ib diamond plate was used as the substrate.

2. Experimental method

The polished substrate was a type-Ib diamond (001) plate provided by Sumitomo Electric Industries, Ltd. The substrate was polished using scaife. To produce a flat surface, the normal to the substrate surface was misoriented from the [001] direction by approximately 3° owing to the step-flow homoepitaxial growth of the substrate surface [14, 15]. The epitaxial layer comprised diamond lightly doped with boron (p^- layer). Such a layer has been used as a semiconducting layer in diamond devices. We assumed that the quality of the epitaxial diamond layer might be dependent on the dislocation density and variations in the distributions of defects in the substrate. Therefore, we used the same substrate throughout the experiment in order to evaluate the effect of the substrate surface flatness on the epitaxial growth of the CVD diamond layer. The scaife-polished surface was subjected to further processing steps described in the next paragraph.

The first step was the ultraviolet-irradiated precision polishing (UV polishing) for an ultraflat finish [16, 17].

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*corresponding author; e-mail: yukako.katou@aist.go.jp

UV polishing was developed in the Touge Laboratory at Kumamoto University, Japan. The method involves the use of a quartz disk and a device that emits ultraviolet radiation. The polishing process is a combination of mechanical polishing with a UV-induced photochemical reaction. The carbon atoms at the surface of the diamond layer are oxidized by active species, such as oxygen radicals, and removed in the form of CO and CO_2 [18]. The surface morphologies were determined using a surface profilometer (Dektak). The mean roughness value, R_a (an arithmetic mean), is a typical parameter used to characterize the roughness of a surface. The R_a of the ultraflat finished surface was about 22 Å. The second step in the process is the p^- layer deposition. The thickness of the p^- layer was 10 μm. The p^- layer was deposited using microwave-plasma-assisted CVD, using hydrogen, methane, carbon dioxide, and trimethyl borate as the source gases. After deposition, the p^- layer was removed by scaife polishing (the third step in the process). The R_a of this surface of the sample was about 70 Å. For the final step, the p^- layer was deposited again, as in the second step.

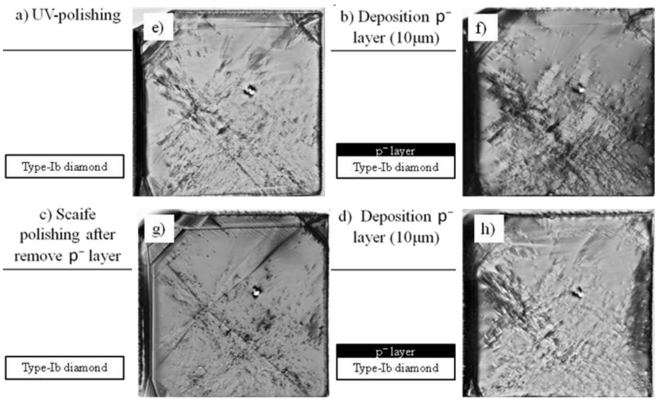

Fig. 1. Schematic view of the experimental method and XRT sample images of each step in the process. For simplicity, a type-Ib diamond plate was used as the substrate for investigating the UV-polishing effect on the quality of the epitaxial diamond layer. (a) UV-polishing, (b) deposition of the p^- layer, (c) scaife polishing after the removal of the p^- layer, and (d) deposition of the p^- layer with same deposition parameters shown in Fig. 1b. After each step in the process, the dislocation distribution was observed using XRT imaging. These XRT images are shown in Fig. 1e–h.

After the each step, the sample was observed by XRT, and the obtained images are shown in Fig. 1e–h. XRT produces two-dimensional images of X-ray diffraction intensities, and these images can provide a distribution map of dislocations in a single crystal [19, 20]. The bright areas represent areas virtually free of all defects. The XRT-based measurements were carried out at beamlines BL14B and BL15C at the Photon Factory in Japan. Conventional monochromatic X-rays from a double-bounce Si (111) crystal monochromator have wavelengths in the range from 0.7 to 1.0 Å. These X-rays were used in our measurements. Nuclear emulsion plates were prepared to capture the XRT images. Because the emulsion particle size was approximately 0.2 μm, this technique was limited to crystals with dislocation densities less than 10^8 cm^{-2} [21].

3. Results and discussion

Using XRT imaging, the dislocation density of the substrate was estimated to be 1.2×10^4 cm^{-2}. After UV-polishing, the dislocation density of the p^- layer was also 1.2×10^4 cm^{-2}. On the other hand, the dislocation density of the p^- layer deposited on the scaife-polished surface was 1.4×10^4 cm^{-2}. In particular, the dislocation density increased remarkably in the upper-right corner of the sample polished by scaife. The dislocation density in this area was 1.9×10^4 cm^{-2}. After UV-polishing, the dislocation density in the same area was 1.2×10^4 cm^{-2}.

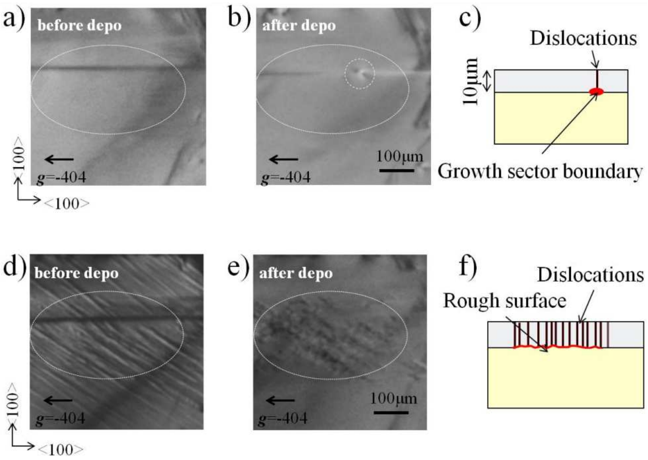

Fig. 2. Magnified XRT images of the upper-right corner of each surface and schematic cross-section views. Figure 2a, b, d, and e are XRT images with a diffraction condition of $g = [-404]$. Figure 2c and f are schematic cross-section views.

To discuss the cause of the differences in dislocation density in the upper-right corner of the sample, magnified images are shown in Fig. 2. Figure 2a, b, d, and e are XRT images with a diffraction condition of $g = [-404]$. Figure 2a is the XRT image of the UV-polished substrate and shows its dislocation distribution. In this area, there is no remarkable dislocation. The horizontal line is the growth sector boundary, which is visible because of topographic contrast [22]. After the p^- layer deposition, one dislocation appeared on this growth sector boundary, as shown in Fig. 2b. The growth direction of the dislocation was $\langle 001 \rangle$, as shown in Fig. 2d.

Figure 2d is the XRT image of the scaife-polished substrate. Many of the scratch marks in Fig. 2d are polishing marks [23]. It is assumed that this defective surface was caused by temporary non-uniform polishing pressure by the scaife. After p^- layer deposition, the dislocation is not as large as in Fig. 2b, but many dislocations appeared on the scratch lines of the substrate, as shown in Fig. 2e. The growth direction of these dislocations was ⟨001⟩, as shown in Fig. 2f. Because these dislocations grew on the rough surface, it is assumed that they stem from the missing step-flow growth at the scratches.

4. Conclusion

A UV-polished substrate reduces the starting points of dislocation growth, because this polishing method is able to produce an ultraflat surface without scratches. According to the experimental data in this study, scratch asperities may disturb the continuous stepping flow growth of the diamond layer. On the other hand, a dislocation is observed in the epitaxial layer on the UV-polished substrate. Because there is no other major defect or defective surface in this area, it is assumed that the strain around the growth sector boundary caused the dislocation in the epitaxial layer on the UV-polished substrate. This indicates that the use of a UV-polished substrate does not eliminate all types of dislocations. However, the density of dislocations caused by scratches from mechanical polishing is decreased in the epitaxial layer.

In this study, we used UV-assisted machining to obtain an ultraflat surface substrate. This method was successful for reducing the starting points of dislocation growth in the epitaxial layer. Another technique for obtaining a smooth surface is CVD growth, which has been reported for the growth of (110) or (111) surfaces [24, 25], but this technique has not been applied to grow (001) surface, which is a typical surface orientation used in diamond devices.

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