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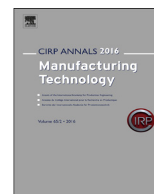

Damage-free highly efficient polishing of single-crystal diamond wafer by plasma-assisted polishing

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ABSTRACT

Single-crystal diamond (SCD) is considered to be an ideal material for next-generation power devices. Plasma-assisted polishing (PAP) without using an abrasive was applied to polish SCD fabricated by chemical vapor deposition. Argon-based plasma containing water vapour was used in the PAP to modify the surface of polishing plate and SCD (100), and SCD was polished under a polishing pressure ranging from 10 to 52.6 kPa. Raman spectroscopy measurement showed that there was no residual stress on the polished SCD surface, and a polishing rate of 2.1 μm/h and a surface roughness of 0.13 nm Sq were obtained.
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1. Introduction

Single-crystal diamond (SCD) is considered to be an ideal material for next-generation semiconductor power devices owing to its excellent electronic and thermal properties such as a wide band gap (5.5 eV), high breakdown field (>10 MV/cm), and high thermal conductivity (2000 W/mK). Typically, large SCD wafers are synthesized by chemical vapor deposition (CVD). At National Institute of Advanced Industrial Science and Technology (AIST) of Japan, a 1-in. SCD wafer was fabricated by a clone method [1]. Since the surface of an SCD wafer after CVD growth is too rough for the wafer to be used as a substrate for power devices, planarizing and smoothing are essential. However, diamond is one of the most difficult-to-machine materials owing to its high hardness and chemical inertness. The method most commonly used to planarize SCD wafers is scaif polishing using diamond abrasives. Unfortunately, scaif polishing has many problems such as a low material removal rate (MRR), the breaking of thin wafers when a polishing load is applied, and the formation of damage on the surface and subsurface [2].

To resolve these issues, a highly efficient and damage-free planarization technique for SCD wafers is strongly required, and some unconventional polishing techniques have been proposed. The tribochemical polishing (TCP) of CVD diamond films using an FeNiCr alloy polishing plate was proposed by Yuan et al., for which as MRR of 3.7 μm/h was obtained [3]. In TCP, the surface of diamond is converted to a non-diamond carbon such as graphite through friction heating and the interaction between the diamond and the catalytic metal polishing plate, then the non-diamond carbon component is removed by mechanical friction, oxidation, and diffuision into the polishing plate. However, compressive residual stress remains on the polished surface as can be detected by Raman scattering spectroscopy. Hocheng and Chen demonstrated the chemical-assisted mechanical polishing of CVD polycrystalline diamond films using different slurries containing diamond powder with a particle size of 4–8 μm and an oxidizing agent. For this method, the highest MRR of 1.39 μm/h was obtaining using potassium persulfate ($K_2S_2O_8$) as the oxidizing agent [4]. Watanabe et al. proposed ultraviolet (UV)-irradiated polishing [5]. The irradiation of UV light with a wavelength rage of 200–400 nm from the rear of a polishing plate made of synthesized quartz glass promoted the removal of the SCD (100) plane, and an MRR of 0.5 μm/h, 1.7 times greater than that without UV irradiation, was obtained. Tatsumi et al. demonstrated the chemical polishing of type IIa SCD (100) using a polishing plate made of quartz glass, and a volumetric MRR of 0.74 mm^3/h, corresponding to an MRR of 27 μm/h in the thickness direction, was obtained [6]. In their proposed removal model, the catalytic oxidation of diamond induced by the friction of quartz glass and/or oxidation of the diamond surface induced by tribological plasma, which was confirmed by optical emission spectroscopy, removes carbon atoms.

On the other hand, we previously proposed a two-step finishing process for a CVD-grown SCD wafer that consists of noncontact dry planarization by numerically controlled plasma chemical vaporization (NC-PCVM) and finishing by plasma assisted polishing (PAP) [7]. In the NC-PCVM process, control of the scanning speed of a localized atmospheric pressure microwave plasma jet improved the flatness of an SCD wafer with a size of 4 mm × 4.2 mm from 7. 2 μm to 3.9 μm in a total processing time of 96 min. Our proposed method of PAP is a novel chemical dry polishing technique that involves modification of the surface by plasma irradiation and removal of the modified surface layer by a soft abrasive. In our previous research, difficult-to-polish wide-gap semiconductor materials such as

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4H-SiC (0001) and GaN (0001) were polished by PAP, and atomically smooth surfaces without the formation of subsurface damage (SSD) and etch pits were obtained [8–11]. In this paper, highly efficient, abrasive-free PAP of a CVD-grown SCD (100) wafer is demonstrated.

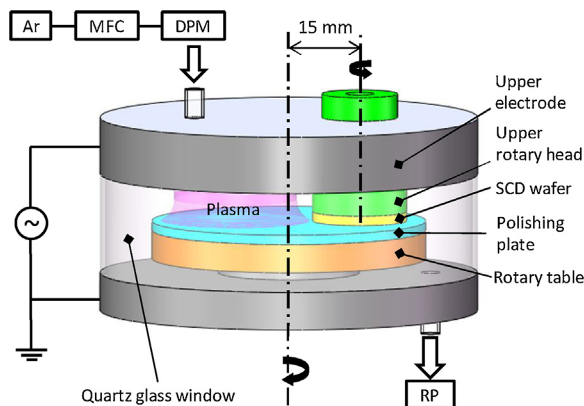

Fig. 1. Schematic images of the experimental setups.

2. Experimental setup

Fig. 1 shows a schematic of the laboratory-built experimental apparatus used in this study. The apparatus consists of upper and lower metal electrodes made of aluminum alloy, and a quartz glass tube to reduce the pressure required for plasma generation between them.

A CVD-grown triangular SCD (100) wafer with an area of 93 mm^2 was set on the upper rotary head, whose center of rotation was offset 15 mm from the center of rotation of the lower rotary table. Argon gas containing water vapor with a concentration of 0.67% was supplied with a flow rate of 100 cm^3/min, and the working gas pressure in the apparatus was 2.3 kPa. The flow rate of Ar was controlled by a mass flow controller (MFC) and the concentration of water vapor was measured by a dew point meter (DPM). A synthesized quartz glass plate (50 mm × 50 mm × 5 mm) was used as a polishing plate. The polishing plate was installed on the lower rotary table and the gap between the upper electrode and the polishing plate was 10 mm. Argon-based plasma was generated at the gap, and the surface of the polishing plate was irradiated by the plasma. The SCD wafer was pressed against the polishing plate with a constant load, and polishing was conducted without using loose or fixed abrasives. Neither the SCD nor the polishing plate was heated. The polishing pressure, the rotation speeds of the polishing plate and SCD wafer, and the supplied RF (f = 13.56 MHz) power were 52.6 kPa, 310 rpm, 20 rpm, and 30 W, respectively. The surface roughness of the SCD was measured by atomic force microscopy (AFM; Shimadzu SPM-9700), and the crystallinity was evaluated by micro-Raman spectroscopy (HORIBA LabRAM HR-800) with a laser spot of size 1 μm.

3. Results and discussion

3.1. Optical emission spectrum from Ar-based plasma

Fig. 2 shows a typical optical emission spectrum (OES) from the Ar-based plasma generated in the experimental apparatus. Optical emission from Ar, OH, H, N_2, and O were observed from the spectrum. It was assumed that OH and H were generated by the dissociation of H_2O molecules remaining in the vacuum vessel and gas supply tube and that N_2 and O_2 entered the vacuum vessel from the outside ambient owing to leakage. Since hydroxyl radicals (OH) have a high oxidation potential, efficient modification of both the surface of the quartz glass plate and the diamond was expected.

3.2. PAP of SCD (100)

Polishing experiments on an SCD (100) wafers were conducted with and without plasma irradiation. The MRR of the SCD was

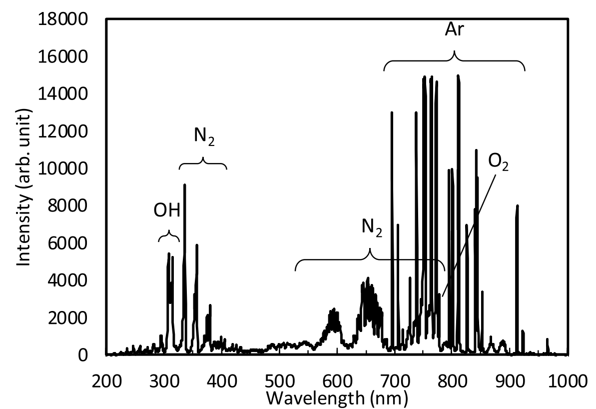

Fig. 2. Optical emission spectrum from Ar based plasma.

calculated from the difference between the removal spot depth before and after polishing, where the removal spot was formed on the SCD wafer surface by atmospheric pressure microwave Ar plasma jet etching. Active Ar radicals excited by the plasma dissociated O_2 molecules in the atmosphere to atomic oxygen, and the atomic oxygen etched the surface of the SCD by converting the carbon atoms to carbon dioxide without forming SSD [7].

In the case of polishing without plasma irradiation, the MRR of the SCD wafer was 0.095 μm/h. In contrast, in the case of PAP with the same experimental parameters, an MRR of 2.1 μm/h, 20 times greater than that without plasma irradiation, was obtained. Fig. 3 shows a large-scale (30 μm × 30 μm) AFM image of the SCD (100) substrate before PAP. Large steps with a height of ca. 350 nm were observed on the CVD-grown SCD surface. These step-bunching structures were formed by the introduction of a small amount of nitrogen in the CVD growth process [12,13] and should be removed before the substrate is used in a power device. Fig. 4(a) and (b) show narrow-scale (5 μm × 5 μm) AFM images (a) before and (b) after PAP. Before PAP, large steps with a height of about 350 nm existed on the SCD surface as shown in Fig. 4(a). After PAP for 1 h, although the step structure was completely removed and an Sq roughness of 0.46 nm was obtained, as shown in Fig. 4(b), scratch-like structures were formed on the processed surface.

To decrease the surface roughness, PAP experiments were conducted at a lower polishing pressure. Fig. 5(a) shows an AFM image (1 μm × 1 μm) of an SCD surface processed by PAP for 3 h using the quartz glass polishing plate with a polishing pressure of 10 kPa. Although a very low Sq roughness of 0.14 nm was obtained, many scratch-like structures were still observed. These scratches were very shallow with a depth of only about 0.5 nm as shown in Fig. 5(b). After PAP, the surface of the quartz glass plate was abraded,

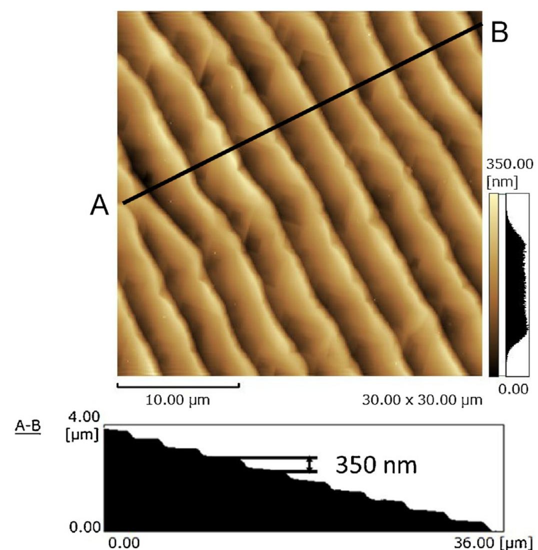

Fig. 3. Step bunching structure of SCD (100) substrate.

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