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Planarization of CVD grown single crystal diamond wafer by numerically controlled plasma chemical vaporization machining

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Abstract

Single crystal diamond (SCD) is considered to be the best material for next-generation power device applications due to its excellent properties. Most of SCD wafers are made by chemical vapor deposition (CVD) and the surface after growth is very rough. Therefore, SCD wafers need planarizing for use in power device material. We proposed numerically controlled plasma chemical vaporization machining (NC-PCVM) using microwave plasma jet etching for planarization. Plasma jet etching generally causes formation of deep etch-pits at dislocation sites of the SCD surface, and the deep etch-pits make polishing time much longer in the following polishing stage. Experimental result shows that deep etch-pits were formed at the surrounding area rather than the center area of the plasma jet. Therefore, we installed an orifice plate to restrict the plasma irradiation area to only the center area of the plasma jet. NC-PCVM planarization with the restriction orifice enabled us to decrease the waviness of the CVD grown SCD wafer without formation of deep etch pits.

NC-PCVM, Atmospheric pressure plasma, Single crystal diamond, Etching, Planarization, Damage-free

1. Background

Single crystal diamond (SCD) is considered to be the best material for next-generation power device applications due to its excellent properties such as a wide band gap (5.5 eV), high break down field (>10 MV/cm). Typically large SCD wafers for electronic device applications are synthesized by chemical vapor deposition (CVD). In national institute of advanced industrial science and technology (AIST) of Japan, SCD wafer with 1-inch size was fabricated by clone method [1]. The surface of SCD wafer after growth by CVD is very rough and it needs planarizing. However, diamond is one of the most difficult-to-machine materials due to its high hardness and chemical inertness. The most popular method to planarize SCD wafers is scife polishing using diamond abrasives. In the case of scife polishing, polishing defects are formed on the surface and subsurface[2]. To resolve these issues, damage-free planarization technique for SCD wafers is strongly required. J. Watanabe *et al*. proposed UV asisted polishing [3]. On the other hand, we proposed numerically controlled plasma chemical vaporization machining (NC-PCVM) using microwave plasma jet [4]. NC-PCVM have been applied to various materials, for example fabrication of elliptical X-ray focusing mirror made of single crystal silicon [5] and correction of thickness distribution of quartz crystal wafer [6]. Meanwhile, it was considered that applying NC-PCVM to SCD wafer is difficult because plasma etching causes formation of deep etch pits at dislocation site of the SCD surface [7]. In our case, deep etch pits were formed at the surrounding area rather than center area of the plasma jet. To resolve this problem, we restricted plasma irradiation area to only the center of the plasma jet by installing an orifice plate. In this paper, positional relationship between plasma jet irradiation area and deep etch-pits formation area are showed, and planarization result of CVD grown SCD (100) wafer by applying NC-PCVM is demonstrated.

2. Experimental setup

NC-PCVM apparatus was consisted of a microwave plasma generator, a sample holder, and 3-axis stage. AlN nozzle with an internal diameter of 0.5 mm was set at the center of the microwave cavity resonator. Ar gas was supplied through the AlN nozzle. Microwave high electric field at the open end of the cavity resonator generated atmospheric pressure Ar plasma jet, and Ar plasma jet generated oxygen radical as the etching species of diamond by dissociation of oxygen molecule in the atmosphere. Sample holder was installed on the 3-axis stage. SCD wafer synthesized by microwave plasma CVD in AIST [1] was planarized by numerically controlled scanning of X-axis and Y-axis. The backside of the SCD wafer was painted to black to increase the absorption efficiency of infrared light, and the wafer was heated by irradiation of infrared light from the backside of the wafer to increase the material removal rate (MRR).

3. Results and discussion

3.1. Positional relationship between plasma jet irradiation area and etch-pits formation area

Plasma etching without scanning of the sample stage was conducted to investigate the detail of formation of etch-pits. Experimental parameters such as flow rate of Ar, microwave power, gap distance between the tip of AlN nozzle and the SCD wafer, surface temperature measured by radiation thermometer without plasma irradiation, and etching time were 0.4 slm, 60 W, 4 mm, 500 °C, and 30 min, respectively.

Fig.1 shows cross-sectional shape of the removal spot and morphologies of the SCD surface after etching measured by scanning white light interferometer (SWLI). In the case of center area of the removal spot (position A in Fig.1), the size and depth of etch pits were small and shallow. In contrast, the size and depth of the etch-pits were large and deep at the outer area of the removal spot where 1.5 mm away from the center of the removal spot (position B in Fig.1). The reason why the difference of size and depth of the etch-pit were occurred was considered as follows. At the center of plasma jet, radical density was high. Therefore, there was little deference of SCD etching rate between defect site and not defect site. On the other hand, radical density at the surrounding of the removal spot was low. Therefore, defect sites were etched preferentially to be large etch-pits. If the deep etch pits are formed, it costs a lot of time to remove deep etch-pits by polishing. So, formation of etch-pits should be avoided. To restrict the etching area to only the center of the plasma jet, orifice plate was installed at the front of the microwave plasma generator as shown in Fig. 2. By installing the orifice plate, formation of deep etch pits at the surrounding of the removal spot were drastically suppressed.

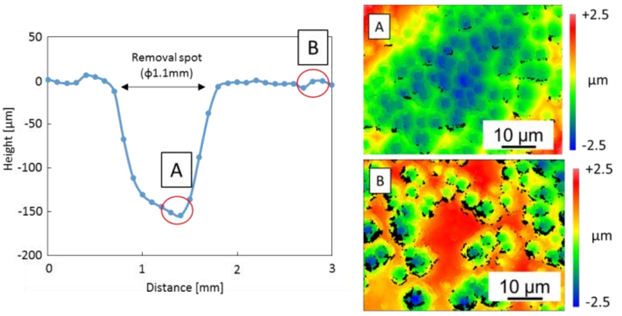


Figure 1. Cross-sectional shape of the removal spot and morphologies of the SCD surface after etching.

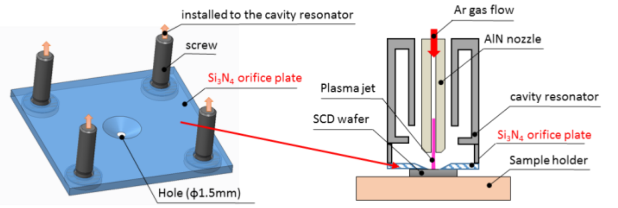
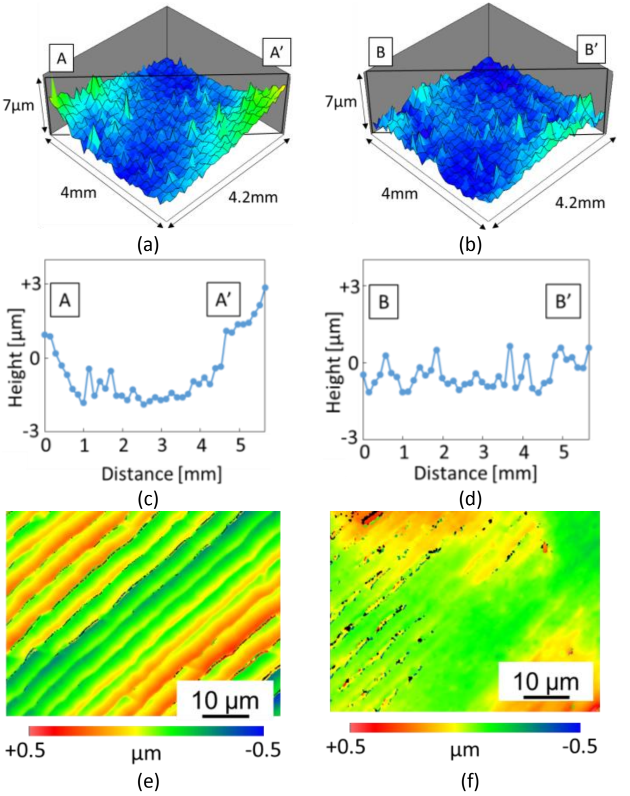


Figure 2. Orifice plate which restrict plasma etching area to only the center of plasma jet.

3.2. *Planarization of SCD wafer by NC-PCVM using microwave plasma jet*

The SCD wafer was planarized by numerically controlled scanning of microwave plasma jet with the orifice. Experimental parameters such as flow rate of Ar, microwave power, gap distance, surface temperature, and processing time were 0.4 slm, 40 W, 4 mm, 500 °C, and 96 min, respectively. Fig.3 shows the three-dimensional shapes and cross-section of the SCD wafer before and after planarization. Shapes of SCD wafer were measured by noncontact laser probe profiler (Mitaka kohki NH3-SP). Measurement pitch was 100 μm. We evaluated 4 mm × 4.2 mm area of the SCD wafer, which excluded the edge of the substrate, because plasma jet was unstable in the edge area of the wafer. By applying NC-PCVM, flatness of the SCD wafer was improved from 7.2 μm to 3.9 μm as shown in Fig. 3(a)(b). In this planarization stage, spatial wavelength component smaller than the diameter of removal spot cannot be removed. Fig.5 (d) shows that the long spatial wavelength component was removed after planarization. Surface morphologies of SCD wafer before and after planarization were measured in detail by SWLI. Fig.3 (e)(f) show that etch-pits were not formed. These results show that NC-PCVM with restriction orifice is pit-free planarization technique. In Fig.3 (e), step-bunching structures, which were formed in CVD synthesis process due to the small amount of nitrogen introduced [8], were observed. Those step-bunching structures were also removed by NC-PCVM as shown Fig. 3 (f).



(a) (b) (c) (d) (e) (f)

Figure 3. Three-dimensional shapes, cross-sections and surface morphologies of SCD wafer (a), (c), (e) before and (b), (d) (f) after planarization.

4. Conclusions

Plasma jet etching generally causes formation of deep etch pits. The etch-pits formed at center of the removal spot were small and shallow, meanwhile the etch pits formed at the surrounding of removal spot were large and deep. We installed the orifice which mask the surrounding of removal spot. Planarization by NC-PCVM using microwave plasma jet with orifice was applied to SCD (100) wafer, and we succeeded in improvement of flatness from 7.2 μm to 3.9 μm with a total processing time of 96 min.

5. Acknowledgments

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References

[1] Yamada H, Chayahara A, Mokuno Y and Tsubouchi N 2011. Diam. Relat. Mater. **20** 616

[2] Volpe P, Muret P, Omnes F, Achard J, Silva F, Brinza O and Gicquel A 2009. Diam. Relat. Mater. **18** 1205

[3] Watanabe J, Mutsumi T and Takeshi S 2013. Diam. Relat. Mater. **39** 14

[4] Dojo H, et al. 2015. Proceedings of ASPEN. 54

[5] Yamamura K, Yamauchi K, mimura H, Sano Y, Saito A, Endo K, Souvorov A, Yabashi M, Tamasaku K, Ishikawa T, and Mori Y 2003. Rev. Sci. Instrum. 74 4549

[6] Yamamura K, Shimada S, Mori Y 2008 Annals of the CIRP **57** 567

[7] Naamoun M, Tallaire A, Silva F, Achard J, Doppelt P and Gicquel A 2012. Phys.Status. Solidi A. **209** 1715

[8] Yamada H, Chayahara A, Mokuno Y. Tsubouchi N and Shikata S 2013. Diam. Relat. Mater. **33** 27