An experimental investigation on high-precision and damage-free processing of diamond

Song Yuan, Benny Chi Fai Cheung*.
State Key Laboratory of Ultra-precision Machining Technology, Department of Industrial and Systems Engineering, The Hong Kong Polytechnic University, Hong Kong, China
Corresponding Author / Email: benny.cheung@polyu.edu.hk

KEYWORDS: CMP, Diamond, Ultra-precision machining, hard and brittle materials

Abstract：*Diamond is honored as the reputation of "ultimate semiconductor material" and the surface with high-precision and damage-free is required for diamond to get the most out of its numerous excellent properties. Chemical mechanical polishing (CMP) is the primary approach to achieve the surface with high-precision and damage-free. However, it is difficult for diamond to be removed and oxidized on account of its properties of large hardness and stable chemical inertness. Herein, the effects of polishing parameters on the surface morphology were investigated by orthogonal experiments. The experiment results indicated that the polishing pressure and the abrasive type are the most significant parameters that determined the surface morphology of diamond. The stronger mechanical force will induce the appearance of pits and scratch on the diamond surface after polishing. However, hydroxyl radicals(•OH) has a favorable promoting effect on improving surface quality of diamond. The surface with high-precision and damage-free can be obtained under the strong mechanical action and •OH. This works sheds lights on the polishing technology for obtaining the diamond with high-precision and damage-free surface.*

1. Introduction

Diamond, as one of the most important "carbon materials", is not only the hardest material in nature but also possesses numerous excellent properties, which is honored as the reputation of "ultimate semiconductor material" and "hardest and sharpest industrial teeth". Before application, the surface with sub-nano level roughness and damage-free is required for diamond to get the most out of it. To obtain the surface with high-precision and damage-free, surface polishing is an indispensable process.

The diamond, known for its exceptional hardness and chemical inertness, poses significant challenges in processing due to its hard and brittle nature. Mechanical processing (MP) currently stands as the primary approach for diamond processing[1]. MP, the conventional method employed, remains the most prevalent technique; however, its reliance on mechanical abrasion by diamond particles results in low processing efficiency and susceptibility to damage[2]. Existing literature indicates that achieving a surface roughness below Ra 0.5 nm through this method is arduous. Though alternative techniques such as laser polishing[3], frictional chemical polishing[4], thermochemical polishing[5], ion beam polishing[6], are plagued by issues like low efficiency, poor post-polishing surface quality, high costs, and demanding environmental equipment requirements. The absence of a mature and robust processing technology tailored for diamond wafers persists. Given diamond's hardness and chemical stability, enhancing its oxidation rate is crucial for improving removal efficiency. Extensive domestic and international research endeavors aim to boost the chemical reactivity of diamond surfaces by diversifying oxidants in polishing fluids, adjusting concentrations, optimizing pressure, temperature, and polishing techniques[7-9]. Chemical mechanical polishing (CMP) emerges as a key approach for achieving high-precision, damage-free surfaces[10]. Nevertheless, the formidable challenges posed by diamond's hardness and chemical stability impede its removal and oxidation, underscoring the persistent difficulty in diamond polishing, a critical bottleneck hindering its broader applications.

Polishing pressure and abrasive type represent the foremost mechanical parameters pivotal for regulating the CMP process. Extensive research has delved into the impact of process variables including pressure and rotational speed on diamond polishing. The Preston equation stands as the predominant model correlating the material removal rate (MRR) with these polishing parameters, illustrating a direct proportionality between the removal rate of surface material and contact pressure as well as relative sliding speed. Empirical evidence demonstrates that heightened pressure and rotational speed can elevate the MRR, albeit at the expense of inflicting significant damage on the processed material. As such, it is imperative to investigate the critical polishing conditions for efficiently polishing diamond surfaces. This study employs orthogonal experiments to scrutinize the effects of key polishing parameters like polishing pressure and abrasive type on the surface quality of polished diamond, ultimately identifying the most optimal polishing parameters for achieving superior outcomes.

2. Experimental setup

Diamond polishing experiments were conducted using various abrasive types and different polishing pressures. On account of the small size of the diamond wafer, three wafers measuring 3 mm × 3 mm × 1 mm were initially embedded in AB resin within a cross section of 5 mm × 5 mm. Subsequently, wax was employed to secure the resin onto the counterweight block. This approach facilitates easy chip disassembly and is convenient for subsequent analysis. Throughout the polishing process, the diamond wafers underwent relative motion with the polishing plate driven by rubbing. The specific polishing parameters are detailed in Table 1. To ensure uniform distribution of abrasive particles, a magnetic stirrer was consistently utilized to agitate the polishing liquid. Moreover, the prepared polishing liquid underwent ultrasonic treatment for 10 minutes prior to polishing to enhance the dispersion of abrasive particles. Surface morphology, surface roughness (Sa), and flatness (PV) values were assessed using a 3D optical surface profiler (Zygo Newview Model) with the measurement area of 868 μm × 868 μm.

Table 1 Polishing parameters

Parameters	Values
Polishing pressures	0.4, 0.6, 1, 1.2 MPa
Polishing speeds	60 r/min
Abrasive concentrations	1 wt%
Abrasive size	W0.25, W0.05
Abrasive type	Diamond, silica
oxidizing agent	30% H_2O_2 aqueous

3. Results and discussion

To achieve a superior surface finish efficiently, larger diamond abrasive particles and increased polishing pressure are selected during the roughing stage. Initially, W10, W5, and W2.5 diamond micro-powders are employed for mechanical polishing of the surface. Following this roughing phase, once the surface roughness stabilizes, CMP is executed to attain an exceptionally smooth surface.

Given its characteristic hardness and brittleness, diamond's removal heavily hinges on polishing pressure. High polishing pressure primarily relies on the abrasive particles' mechanical force, leading to a brittle removal mechanism. After a 3-hour polishing session under the conditions of 1 MPa polishing pressure, utilizing W2.5 diamond abrasive with a mass fraction of 1 wt%, the surface morphology of the polished diamond was examined using a 3D optical surface profiler. Observations revealed an increased presence of pits, scratches, and other forms of damage on the surface, culminating in poor surface quality post-processing, as depicted in Fig. 1.

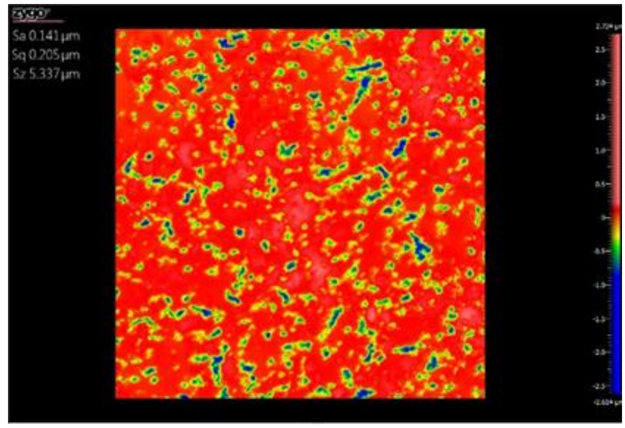

Fig. 1 Surface morphology after MP using diamond abrasive and deionized water

Through the assessment of diamond's surface roughness and MRR at varying polishing pressures, it becomes evident that MRR progressively rises with increasing polishing pressures. Notably, elevating the pressure from 0.4 MPa to 0.6 MPa results in a substantial MRR boost, while Sa remains relatively unchanged, indicating that 0.4 MPa is a low polishing pressure for diamond. Conversely, a pressure escalation from 1 MPa to 1.2 MPa leads to a significant increase in both MRR and Sa, signifying a pronounced effect on the material removal and surface roughness.

Fig. 2 and Fig. 3 depict the surface morphology post-polishing using diamond abrasive with H_2O_2 aqueous and silica abrasive with H_2O_2 aqueous, respectively. Fig. 2 illustrates a notably enhanced surface morphology of polished diamond compared to mechanical polishing with large abrasive particles. After a 1-hour polishing session with small abrasive size diamond abrasive and H_2O_2 aqueous, an ultra-smooth surface roughness of Sa=0.135 nm is swiftly achieved, albeit with evident scratches, pits, and other defects leading to a surface roughness of Sz=68.414 nm.

In the presence of H_2O_2 aqueous, the generated free hydroxyl (•OH) species oxidize the diamond surface, transforming the carbon atoms into a softer oxide layer. The combined mechanical shear force of the diamond abrasive and the chemical action of the oxidizer significantly enhance the surface quality of the polished diamond, albeit with some residual pits due to the mild oxidation effect of H_2O_2 aqueous. The gradual and incomplete oxidation in certain areas results in a removal mechanism primarily driven by oxidation processes.

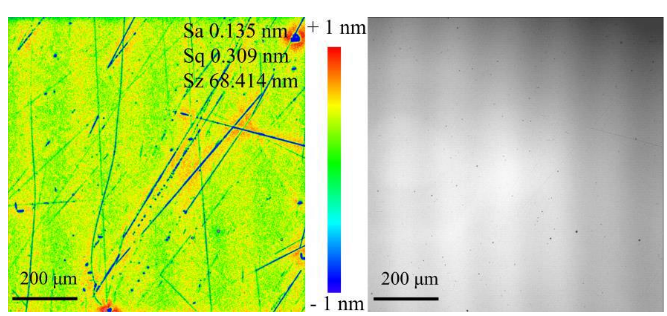

Fig. 2 Surface morphology after CMP using diamond abrasive and H_2O_2 aqueous (Sa=0.135 nm, Sq 0.309 nm, Sz=68.414 nm)

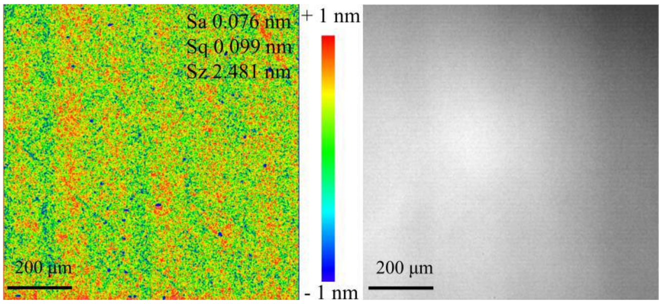

Fig. 3 Surface morphology after CMP using silica abrasive and H_2O_2 aqueous (Sa=0.076 nm, Sq 0.099 nm, Sz=2.481 nm)

Fig. 3 illustrates that under identical process conditions, polishing for 6 hours with silica abrasive and H_2O_2 aqueous resulted in a significantly reduced surface roughness of Sa=0.076 nm, while ensuring a defect-free polished surface without scratches, pits, or other imperfections, yielding a surface roughness of Sz=2.481 nm. These experiments highlight that, given specific conditions, diamond abrasive can achieve superior polishing efficiency, albeit with the drawback of scratches and pits, whereas employing silica abrasive leads to a surface free from subsurface damage at the expense of a lower removal rate.

3. Conclusions

In this study, CMP experiments were conducted on diamond using both diamond abrasive and silica abrasive particles. The research aimed to investigate the variances in diamond removal mechanisms under different abrasive particles, analyze the factors contributing to the low diamond removal rate in H_2O_2 aqueous, and assess the damage induced by diamond abrasive. The key conclusions drawn from the study are as follows:

Using diamond abrasive polishing: scratches and pits are observed on the diamond surface post-polishing with diamond abrasive. However, the removal efficiency is notably high. The strong mechanical action of diamond particles results in efficient material removal. The chemical action of H_2O_2 aqueous is relatively weak, leading to a lack of oxidation of C atoms within the matrix, thereby facilitating high removal rates alongside significant damage.

Using silica abrasive polishing: polishing with silica abrasive yields a smooth diamond surface without subsurface damage. However, the removal efficiency is comparatively low. The weak mechanical action of silica abrasive contributes to a slower removal process. Diamond removal occurs in the form of individual C atoms or carbon chains, resulting in minimal damage and a reduced removal rate.

These findings underscore the trade-offs between removal efficiency, surface quality, and damage levels associated with different abrasive particles during diamond polishing. Diamond abrasive offers high removal rates at the expense of surface imperfections, while silica abrasive ensures a pristine surface but with a slower removal rate due to its milder mechanical action.

ACKNOWLEDGEMENT

The authors would like to express their sincerely thanks to the financial support of the Postdoc Matching Fund Scheme from the Research and Innovation Office of The Hong Kong Polytechnic University (Project codes: 1-W29X).

REFERENCES

[1] N. Yang, W. Zong, Z. Li, et al., Wear process of single crystal diamond affected by sliding velocity and contact pressure in mechanical polishing, Diamond and Related Materials, 58 (2015)46-53.

[2] M. Doronin, S. Polyakov, K. Kravchuk, et al., Limits of single crystal diamond surface mechanical polishing, Diamond and Related Materials, 87 (2018)149-155.

[3] R. Yoshizaki, M. Nakao, Ultrashort pulse laser processing of single crystalline diamond for efficient and smooth grooving with top-hat beam modulation, CIRP Annals, 72 (2023)189-192.

[4] T. Yang, D. Song, Z. Hao, Progress and prospect of diamond dynamic friction polishing technology, The International Journal of Advanced Manufacturing Technology, 124 (2023)1357-1378.

[5] Y. Imoto, J. Yan, Thermochemical micro imprinting of single-crystal diamond surface using a nickel mold under high-pressure conditions, Applied Surface Science, 404 (2017)318-325.

[6] S. Mi, A. Toros, T. Graziosi, et al., Non-contact polishing of single crystal diamond by ion beam etching, Diamond and Related Materials, 92 (2019)248-252.

[7] Z. Yuan, Z. Jin, Y. Zhang, et al., Chemical mechanical polishing slurries for chemically vapor-deposited diamond films, Journal of Manufacturing Science and Engineering, 135 (2013)041006.

[8] Y. Lin, J. Lu, R. Tong, et al., Surface damage of single-crystal diamond (100) processed based on a sol-gel polishing tool, Diamond and Related Materials, 83 (2018)46-53.

[9] A. Kubota, S. Nagae, M. Touge, Improvement of material removal rate of single-crystal diamond by polishing using H2O2 solution, Diamond and Related Materials, 70 (2016)39-45.

[10] E.L. Thomas, G.W. Nelson, S. Mandal, et al., Chemical mechanical polishing of thin film diamond, Carbon, 68 (2014)473-479.