Material removal rate of 6H-SiC crystal substrate CMP using an alumina (Al_2O_3) abrasive*

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Abstract: The influences of the polishing slurry composition, such as the pH value, the abrasive size and its concentration, the dispersant and the oxidants, the rotational velocity of the polishing platen and the carrier and the polishing pressure, on the material removal rate of SiC crystal substrate (0001) Si and a (0001) C surface have been studied based on the alumina abrasive in chemical mechanical polishing (CMP). The results proposed by our research here will provide a reference for developing the slurry, optimizing the process parameters, and investigating the material removal mechanism in the CMP of SiC crystal substrate.

Key words: SiC crystal substrate; alumina abrasive; chemical mechanical polishing; material removal rate; polishing slurry

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1. Introduction

Semiconductor lighting is one of the most promising high-tech fields in the 21st century. At the heart of an LED (light emitting diode) is a semiconductor chip. The core technology of an LED chip is manufacturing the epitaxial GaN-based wafer on the substrate. There are many materials used for GaN-based LED substrates, but only two materials, sapphire (Al_2O_3) and silicon carbide (SiC), are currently used for commercial substrate materials. SiC single crystal substrate has been become an indispensable substrate material in the field of semiconductor lighting. LEDs based on a SiC substrate takes the second place in LED market. Therefore, the preparation of substrates is the core technology of the semiconductor lighting industry[1−4].

SiC has been recently applied in the semiconductor industry and optical components due to its high hardness, excellent thermal conductivity, good chemical stability, the unique wide-band-gap, high critical puncture electric intensity, high electron mobility, etc. In recent years, SiC has rapidly gained interest as a substrate for the fabrication of epitaxial devices[5]. Nowadays, SiC single crystals have been become the ideal substrate material for manufacturing optoelectronic integrated devices, such as high-temperature, high-frequency, high power, anti-radiation and short wavelength light-emitting components, and is also a research focus in the fields of semiconductors, microelectronics, and optoelectronics[6].

Research shows that device quality largely depends on surface quality. Because the substrate surface quality has a great impact on epitaxial films. The high surface quality of SiC substrates is very difficult to obtain due to its high hardness and high chemical stability. Chemical mechanical polishing (CMP) is one of the core technologies used in the field of semiconductor manufacturing and has been become the most widely used technology in ULSI manufacturing. Therefore, CMP is the most effective technology used for achieving ultra-smooth undamaged surfaces in the ultra-precision machining of SiC crystal substrates[5−9].

Some researchers have studied the CMP of a SiC substrate using different abrasives and methods. An *et al.*[3] have conducted a CMP test of 6H-SiC substrate using a diamond abrasive and silica sol, respectively. Neslen *et al.*[4] studied the CMP of 4H-SiC using the silica sol abrasive. Lee *et al.*[5, 8, 9] studied the CMP of 6H-SiC substrate with a mixed abrasive of diamond abrasive and silica sol. Lin *et al.*[6] studied the surface polishing of SiC with a tribochemical reaction mechanism. Hara *et al.*[7] have proposed a planarization method of catalytic-referred etching and conducted the planarization test of 4H-SiC using a solid Pt polishing pad. The hardness of the abrasive alumina (Al_2O_3) is lower than that of SiC substrate and abrasive diamond, but the price of the abrasive alumina is much lower than that of abrasive diamond. Up to now, a report studying SiC substrate CMP based on an alumina abrasive has not been found.

In this paper, a series of CMP tests of 6H-SiC (0001) C and (0001) Si surfaces have been conducted using abrasive alumina (Al_2O_3) slurry. The influences of the abrasive content, the chemical composition, polishing velocity and polishing pressure on the material removal rate (MRR) have been

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Table 1. The basic slurry composition from orthogonal design experiments.

Ingredient	pH value	Oxidant content (mL/500 mL)	Dispersant content (mL/500 mL)	Abrasive size (μm)	Abrasive content (g/500 mL)	Others
Si surface	9	5	2	1.5	10	DI water
C surface	9	5	4	3.5	10	DI water

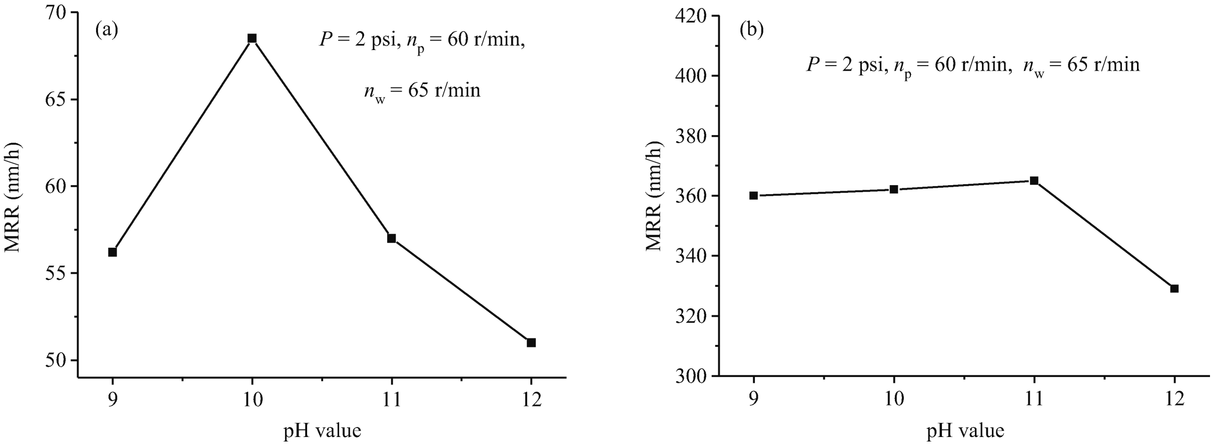

Fig. 1. The influence of the pH value on the MRR. (a) (0001) Si surface. (b) (0001) C surface.

studied. It is hoped that our results will provide a reference for reducing the cost of SiC crystal substrate CMP.

2. Experiments

The CMP experiments were conducted under different parameters and slurry on the ZYP300 type CMP machine produced by Shenyan. All the experiments are done in a clean room with Grade 1000 at a constant temperature of 22 ℃. Many 6H-SiC wafers of 2 inches in diameter are used in the polishing experiments. Samples are bonded with paraffin on a stainless steel carrier of Φ115 mm in diameter. Each carrier has one SiC wafer stuck onto it. The roughness of the (0001) Si and C surface before polishing is about Ra 0.403 μm, which is tested by using a Talysurf CCI 3D surface profiler. In the CMP experiments, a polyurethane pad is applied and conditioned for 15 min before every polishing experiment with a diamond conditioner. The slurry is supplied at a flow rate of 15 mL/min. The slurry (500 mL) can be made before each test. During the CMP process, the carrier has a reciprocating motion with a stroke of 20 mm at a frequency of 10 s and the centre distance between the pad and the wafer is set as 80 mm. The polishing time is 30 min for each test. Deionized (DI) water with an electrical resistivity of 18.24 MΩ·cm is used in the CMP experiments. The MRR can be calculated by weighing the SiC wafer on a high precision balance with accuracy of 0.01 mg before and after CMP.

3. Experimental results and analysis

Because of the difference of the chemical and physical properties between the SiC (0001) C surface and the Si surface, the slurry composition will not be the same. According to the previous research and the orthogonal design method, orthogonal design CMP experiments of SiC (0001) C surface and Si surface have been conducted. The basic slurry compositions for SiC (0001) C surface and Si surface CMP have been obtained respectively, as shown in Table 1. Then, according to the basic slurry composition, the influence of the slurry composition and other parameters on MRR has been found by changing various parameters.

3.1. The influence of pH value on the MRR

From the literature, CMP of hard and brittle materials is carried out in an alkaline environment with the pH value of the polishing slurry between 9 and 13[8], so the pH value of slurry is selected between 9 and 13 in experiments. The pH value is adjusted with alkali. The experiment parameters are: polishing pressure, $P = 2$ psi; rotational velocity of the carrier, $n_{\rm w} = 65$ r/min; and rotational velocity of the polishing platen, $n_{\rm p} = 60$ r/min. The MRR curve with the pH value is shown in Fig. 1.

By Figs. 1(a) and 1(b), the MRR increases with the increase of pH value at the beginning. After reaching a certain value, the MRR decreases with the increase of the pH value. It is indicated that there is an optimum pH value at the point of the maximum of MRR. Also it shows that it can promote the chemical reaction in an alkaline environment. In the CMP process, frictional heat can be produced on the polishing interface between the SiC wafer and the polishing pad, which can promote the alkali reaction with the surface material of the SiC substrate at a certain temperatures[10]. The silicate can be generated when the SiC reacts with alkali under a certain pressure and temperature. The reaction process can be controlled by the pH value. By adjusting the pH value, the reaction speed changes, thereby changing the MRR.

Comparing Fig. 1(a) with Fig. 1(b), the optimum pH value is 10 in Si surface CMP and 11 in C surface CMP. The MRR of C surface CMP is larger than that of Si surface CMP.

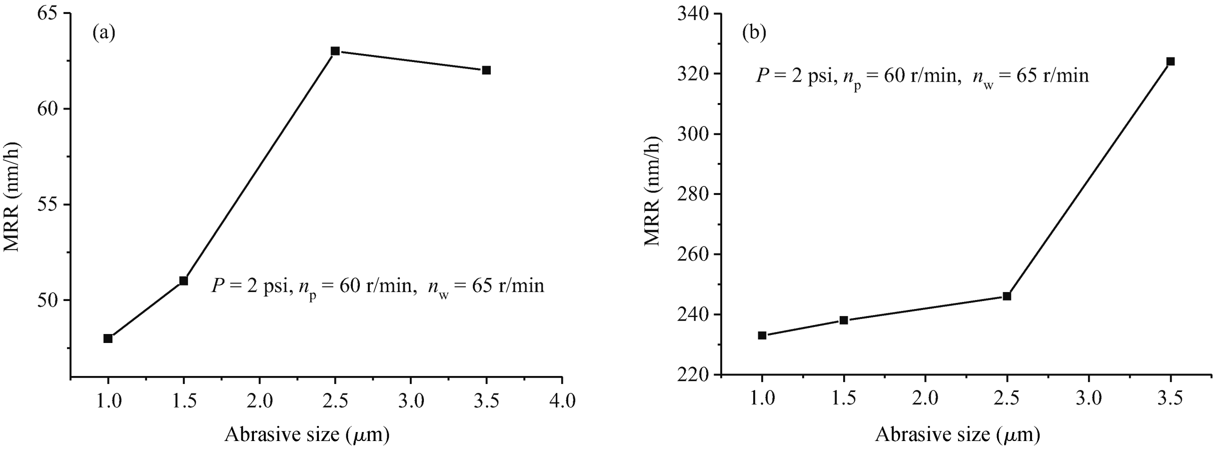

Fig. 2. The influence of abrasive size on the MRR. (a) (0001) Si surface. (b) (0001) C surface.

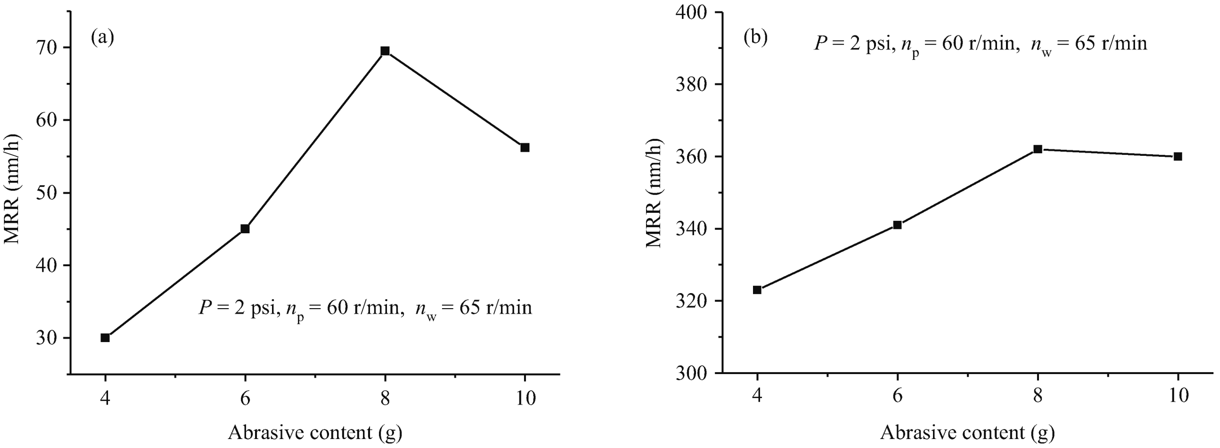

Fig. 3. The influence of abrasive content on MRR. (a) (0001) Si surface. (b) (0001) C surface.

3.2. The influence of abrasive size on the MRR

Four abrasive sizes of 1 μm, 1.5 μm, 2.5 μm, 3.5 μm are used in this study. The experimental parameters are $P = 2$ psi, $n_{\rm w} = 65$ r/min, $n_{\rm p} = 60$ r/min. The curve of the MRR with the abrasive size is shown in Fig. 2.

Figure 2 shows that the MRR increases with the increase of the abrasive size. The mechanical action on the SiC surface is enhanced with the abrasive size, thereby increasing the MRR.

Comparing Fig. 2(a) with Fig. 2(b), the optimum abrasive size is about 2.5 μm for Si surface and 3.5 μm for C surface in CMP respectively. The MRR of C surface CMP also is about 5 times larger than that of Si surface CMP.

3.3. The influence of abrasive content on the MRR

The abrasive content will also have an impact on the CMP MRR. Take the four abrasive contents 4 g, 6 g, 8 g, 10 g, with the abrasive size 1.5 μm for CMP of a Si surface and with the abrasive size 3.5 μm for CMP of a C surface. The experiment parameters are $P = 2$ psi, $n_{\rm w} = 65$ r/min, $n_{\rm p} = 60$ r/min. The curve of MRR with the abrasive content is shown in Fig. 3.

From Figs. 3(a) and 3(b), the MRR increases with the increase of the abrasive concentration. When the abrasive content increases to 10 g, the MRR reduces to 56.2 nm/h and 360 nm/h, respectively. This is mainly because that the amount of abrasive involved in the mechanical action increases with the increase of the abrasive concentration at the beginning. It makes the mechanical action of the abrasive also increase during the polishing process, thereby increasing the MRR. However, with the further increase of the abrasive concentration, the MRR decreases slightly. Because the diameter of the abrasive is very small, a high content of abrasive in the slurry leads to it having a large density. Therefore, the fluidity of the slurry decreases and causes the chemical reaction to be relatively slow as compared to the increase of the mechanical action, so, the MRR has declined.

Comparing Fig. 3(a) with Fig. 3(b), the optimum abrasive content in 500 mL of slurry is about 8 g in Si and C surface CMP. The MRR of C surface CMP also is about 5 times larger than that of CMP Si surface.

3.4. The influence of dispersant content on the MRR

The role of the dispersant is to make the abrasive disperse in the slurry uniformly. Its content can affect the dispersion degree of the abrasive in the polishing slurry. Therefore, the dispersion degree of an abrasive will also impact the MRR. Now, take benzyl alcohol as the dispersant under different contents for studying the MRR. Four slurries are prepared with different dispersant contents, 2 mL, 8 mL, 14 mL, and 20 mL, in 500

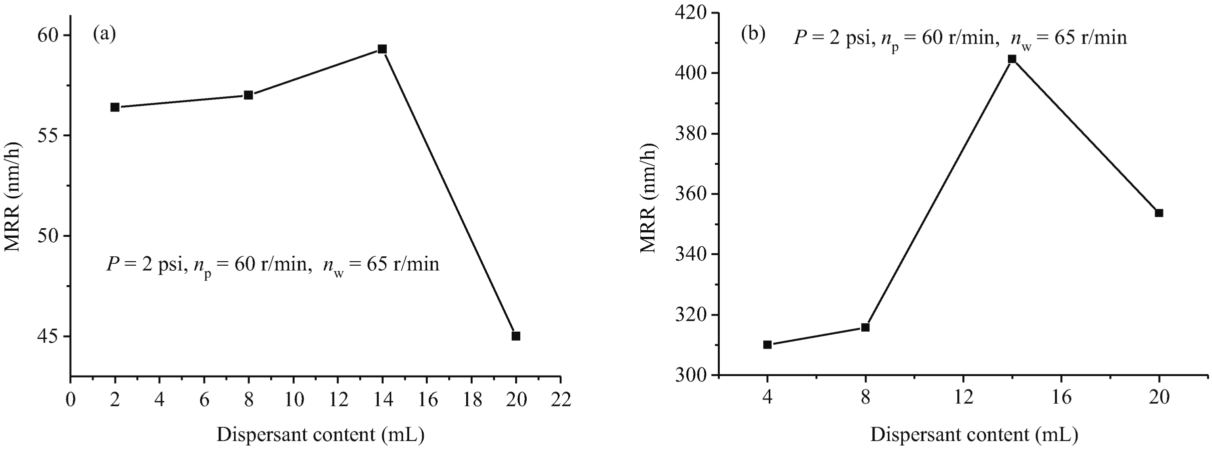

Fig. 4. The influence of dispersant content on MRR. (a) (0001) Si surface. (b) (0001) C surface.

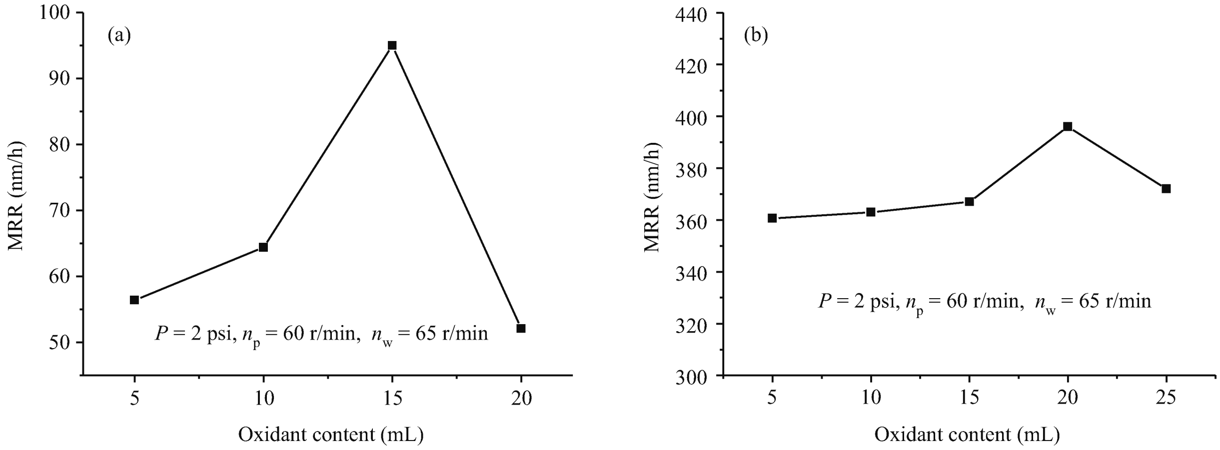

Fig. 5. The influence of oxidant content on MRR. (a) (0001) Si surface. (b) (0001) C surface.

mL of slurry for Si surface CMP, respectively. Four slurries are also prepared with different dispersant contents, 4 mL, 8 mL, 14 mL, and 20 mL, in slurry 500 mL for C surface CMP, respectively. The experimental parameters are $P = 2$ psi, $n_{\rm w} =$ 65 r/min, $n_{\rm p} = 60$ r/min. The experimental results are shown in Fig. 4.

Figures 4(a) and 4(b) show that the MRR increases with the increase of the dispersant concentration when the dispersant content is low. When the dispersant content is 14 mL, the MRRs reach maximums of 59.3 nm/h and 404.5 nm/h, respectively. When the dispersant content is increased to 20 mL, however, the MRR reduces to 45 nm/h and 353.5 nm/h, respectively. This is mainly because the dispersion degree of the abrasive increases with the increase of the dispersant concentration in slurry when the dispersant concentration is low, therefore making the amount of the abrasive involved in the mechanical action increase and the MRR increase. However, when further increasing the dispersant concentration, the MRR decreases slightly, because benzyl alcohol is often used as a lubricant in industry. When its content increases, the lubricity of the slurry may increase, therefore the friction will decrease between the polishing pad and the SiC substrate, and so the MRR decreases. The reason for this will be studied further.

Comparing Fig. 4(a) with Fig. 4(b), the optimum dispersant content in 500 mL of slurry is about 14 mL in Si and C surface CMP. The MRR of C surface CMP also is about 5 times larger than that of Si surface CMP.

3.5. The influence of oxidant content on the MRR

The role of the oxidant is to promote and participate in the chemical reaction with the SiC surface material and generate reactants that can be removed easily on the SiC surface. The reactant will be removed by the mechanical action of the abrasive. Therefore, the oxidant content will change the chemical reaction rate in CMP. We take different amounts of the common oxidant of H_2O_2 for studying the MRR. Slurries are prepared with the different oxidant content, such as 5 mL, 10 mL, 15 mL, and 20 mL, respectively, in 500 mL of slurry. The experimental parameters are $P = 2$ psi, $n_{\rm w} = 65$ r/min, $n_{\rm p} = 60$ r/min. The experimental results are shown in Fig. 5.

By Fig. 5(a), the MRR increases with the increase of the oxidant concentration at the beginning. When the oxidant content is 15 mL, the MRR obtains maximum 95 nm/h. When the oxidant content is 20 mL, the MRR decreases to 52.1 nm/h. Figure 5(b) shows that the MRR increases with the increase of the oxidant concentration at the beginning. When the oxidant content is 20 mL, the MRR obtains a maximum of 395.7 nm/h. The above indicates that there is an optimum oxidant content which can make the MRR reach a maximum. Because CMP is

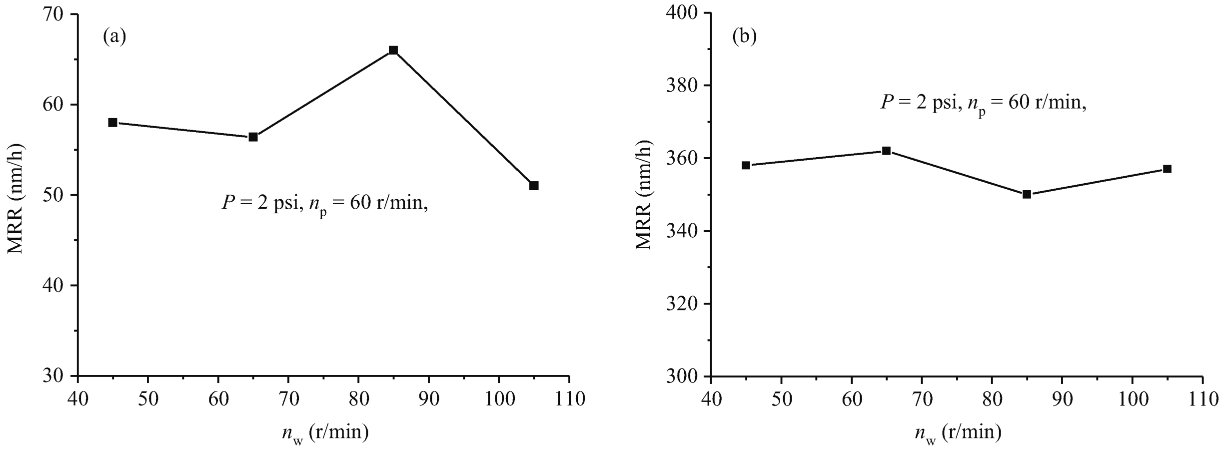

Fig. 6. The influence of n_w on MRR. (a) (0001) Si surface. (b) (0001) C surface.

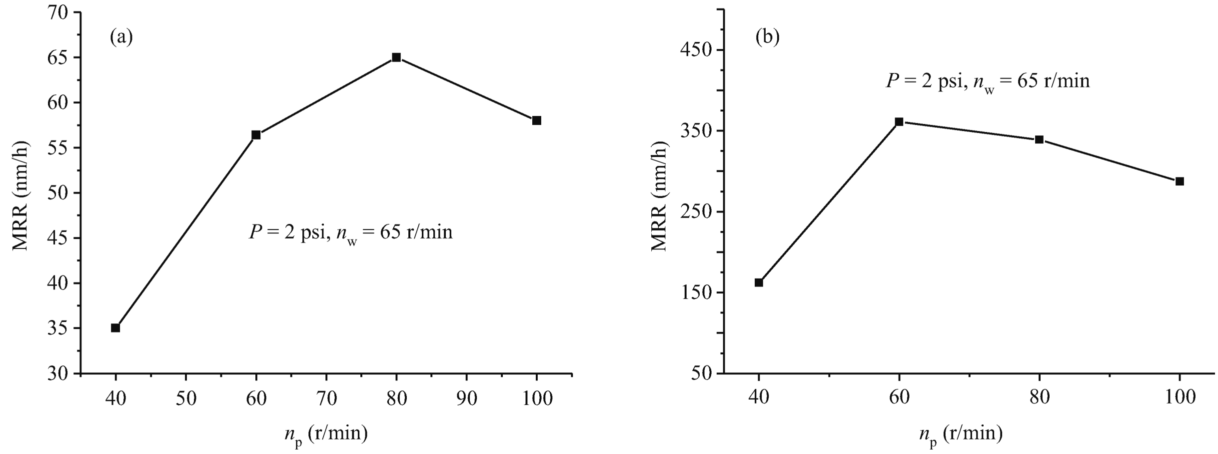

Fig. 7. The influence of n_p on MRR. (a) (0001) Si surface. (b) (0001) C surface.

a process of combining chemical and mechanical actions, only when the chemical action matches the mechanical action, can it achieve a high MRR in CMP.

Comparing Fig. 5(a) with Fig. 5(b), the optimum oxidant content in 500 mL of slurry is about 15 mL in Si CMP and the optimum oxidant content in 500 mL of slurry is about 20 mL in C surface CMP. The MRR of C surface CMP is about 4 times larger than that of Si surface CMP.

3.6. The influence of polishing velocity on MRR

Under the same conditions, a change of the rotational velocity of the polishing platen and the carrier will change the mechanical action of polishing system, so it will affect the MRR. The experimental parameter is $P = 2$ psi. The experiment can be conducted under different rotational velocities of the polishing platen and the polishing carrier, respectively. The experimental results are shown in Figs. 6 and 7.

By Figs. 6(a) and 6(b), when $n_{\rm p} = 60$ r/min, the MRR changes little with the increase of the carrier rotational velocity $n_{\rm w}$. It is indicated that the change of the rotational velocity of the carrier causes little change of the mechanical action. Therefore, it has little effect on the MRR.

By Fig. 7(a), the maximum MRR is about 65 nm/h in Si surface CMP, which occurs at $n_{\rm p} = 80$ r/min, $n_{\rm w} = 65$ r/min. When the rotational velocity of the platen, $n_{\rm p}$, was below 80 r/min, the MRR increased with the increase of rotational velocity $n_{\rm p}$. When the rotational velocity of the platen, $n_{\rm p}$, was above 80 r/min, the MRR decreased with the increase of rotational velocity $n_{\rm p}$. By Fig. 7(b), the maximum MRR is about 360 nm/h in C surface CMP, which occurs at $n_{\rm p} = 60$ r/min, $n_{\rm w} = 65$ r/min. When the rotational velocity of the platen, $n_{\rm p}$, was below 60 r/min, the MRR increased with the increase of rotational velocity $n_{\rm p}$. When rotational velocity of the platen, $n_{\rm p}$, was above 60 r/min, the MRR decreased with the increase of rotational velocity $n_{\rm p}$. Because that CMP is an interaction process between a mechanical action and a chemical action, only when the mechanical action is in balance with the chemical action can the optimal MRR occur.

Comparing Fig. 7(a) with Fig. 7(b), under this experimental condition, it is indicated that the mechanical action is in balance with the chemical action when the rotational velocity of the platen, $n_{\rm p}$, is 80 r/min in Si surface CMP and 60 r/min in C surface CMP and at this point the MRR reaches a maximum.

3.7. The influence of polishing pressure on the MRR

Polishing pressure is an important factor that affects the MRR of SiC CMP. The change of the polishing pressure will change the mechanical action, the chemical action, and the in-

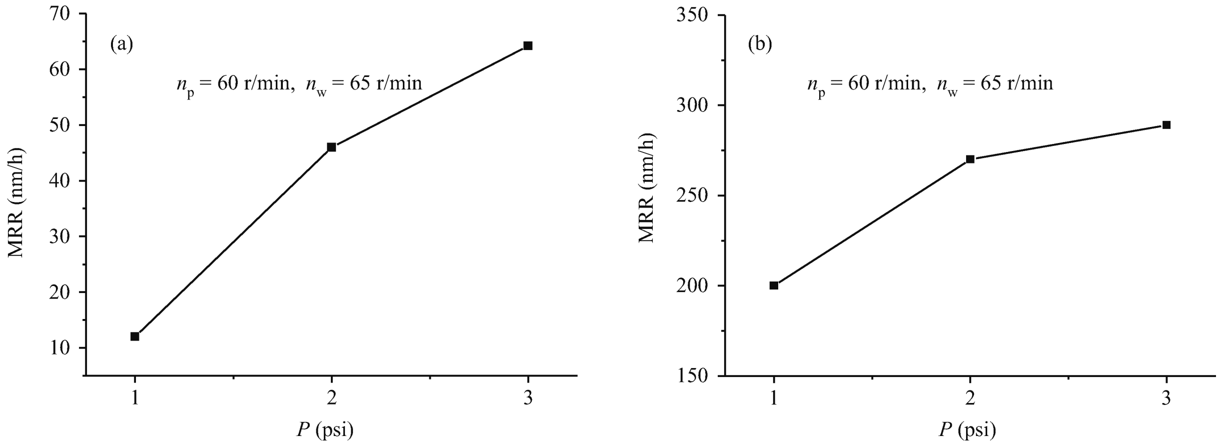

Fig. 8. The influence of polishing pressure on MRR. (a) (0001) Si surface. (b) (0001) C surface.

Table 2. The optimal slurry composition from experimental results and analysis.

Ingredient	pH value	Oxidant content (mL/500 mL)	Dispersant content (mL/500 mL)	Abrasive size (μm)	Abrasive content (g/500 mL)	others
Si surface	10	15	14	2.5	8	DI water
C surface	11	20	14	3.5	8	DI water

teraction of the polishing system, so it will affect the MRR of the wafer surface. The experiment parameters are n_w = 65 r/min, n_p = 60 r/min. The experiment is conducted under different polishing pressures. The experimental results are shown in Fig. 8.

Figure 8 shows that the MRR increases with the increase of the polishing pressure when other conditions are unchanged, the MRR is basically proportional to the polishing pressure, and the curve of the MRR is consistent with the classic Preston equation.

4. Results

According to a series of experimental results and analysis, the influence of various factors on MRR can be obtained in the CMP of a SiC substrate. The optimal slurry composition also can be obtained, see Table 2. By Table 2, several CMP experiments have been done using the optimal slurry composition. The MRR is about 1236 nm/h in Si surface CMP at $P = 2$ psi, $n_w = 65$ r/min, $n_p = 80$ r/min, and the MRR is about 1632 nm/h in C surface CMP at $P = 2$ psi, $n_w = 65$ r/min, $n_p = 60$ r/min. Thus, only when the chemical action matches with the mechanical action, can the higher MRR be obtained. The SiC surfaces, whether the Si surface or the C surface, have no scratches, as observed under a microscope at 1000 times magnification, after CMP. The surface roughness, detected by using a 3D surface profiler, can be up to Ra 1 nm or less after 2.5 h polishing in the experiment. It shows that abrasive alumina (Al_2O_3) can be used in the CMP of SiC crystal substrates.

5. Conclusion

Following analysis of the experimental results, the influence of various factors on the MRR of a SiC substrate surface was obtained.

(1) The CMP slurry should have an optimum pH value, abrasive content, and dispersant content.

(2) The CMP is a combined mechanical and chemical process. Only when the mechanical action is in balance with the chemical action, can the optimal MRR occur.

(3) The rotational velocity of the platen has a greater impact on the MRR, but the rotational velocity of the carrier has a less impact on the MRR.

(4) The chemical reaction rate increases with the increase of the oxidant contents. As long as there is sufficient mechanical action, the MRR will increase with the increase of the oxidant contents.

(5) The polishing pressure has a greater impact on the MRR. The MRR increases with the increase of the polishing pressure under the experimental conditions.

(6) The MRR of C surface CMP is larger than that of Si surface CMP in all of the above experiments, and the MRR when using abrasive alumina is lower than that of CMP using abrasive diamond.

(7) The SiC surfaces, whether the Si surface or the C surface, have no scratches after CMP. The surface roughness can be up to Ra 1 nm or less after a 2.5 h polishing experiment. It is indicated that abrasive alumina (Al_2O_3) can be used in the CMP of SiC crystal substrate. The results will provide a reference for developing the slurry, optimizing the process parameters, and investigating the material removal mechanism in the CMP of SiC crystal substrate.

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