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Experimental study on plasma enhanced electrochemical mechanical polishing of SiC single crystal

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Abstract. SiC single crystal is a good semiconductor material with high breakdown electric field strength, high thermal conductivity, high carrier saturation drift speed and low relative dielectric. However, SiC has high hardness and brittleness, high chemical stability, so the polishing efficiency of existing polishing methods is low. In this paper, a plasma enhanced electrochemical mechanical polishing method for SiC was proposed. The relationship between peak voltage, frequency and duty ratio with polishing efficiency and surface roughness was studied.

Keywords: SiC single crystal; Plasma enhanced electrochemical mechanical polishing; Polishing efficiency; Surface roughness.

1. Introduction

Because of the special physical and chemical properties, semiconductor materials have become one of the indispensable structural materials in electronics industry, photovoltaic industry and aerospace industry [1, 2]. As the representative of third-generation wide band gap semiconductor materials, SiC has the characteristics of large band gap width (2.2~3.3 eV), high thermal conductivity (4.9 W/cm˙K), high critical breakdown electric field (1.5~3.2×106 V/cm) and so on. In high power, high voltage, high temperature, high radiation and other extreme conditions, SiC materials can still show irreplaceable excellent characteristics, which also promote SiC materials has a pivotal position and a wide range of application prospects in the field of semiconductor devices. Due to the high hardness and stable chemical properties of SiC, chemical residues, poor surface quality and low polishing efficiency are easy to occur when using traditional polishing methods.

In recent years, in order to improve the material removal rate (MRR) and surface quality of SiC surface polishing, based on the chemical reaction of SiC precision polishing technology has been widely studied. For example, chemical mechanical polishing (CMP) [3], plasma-assisted polishing (PAP) [4], photocatalytic chemical mechanical polishing (PCMP) [5] and electrochemical mechanical polishing (ECMP) [6, 7]. The common feature is that the SiC surface is first modified to an oxide layer with lower hardness than the SiC substrate, then removed the oxide layer by a softer abrasive machine to obtain a smooth SiC surface. In general, these technologies cannot satisfy the requirements of high efficiency, low cost, no damage and high precision polishing simultaneously.

The traditional discharge machining is to remove the material through the high temperature and electrical erosion, which caused by continuous and rapid discharge between the poles, that immersed in the insulating medium, without any requirement on the brittleness and hardness of the material [8, 9]. However, the surface roughness of the machined workpiece is poor due to the thermal explosion generated in the process of discharge machining. Plasma enhanced electrochemical mechanical polishing (PEECMP) of SiC refers to application of high frequency voltage between the negative and positive poles in water-based solution, to generate a large amount of oxygen on the surface of the anode workpiece (SiC), forming a very high density oxygen gap film, pulse high voltage makes the both ends of gas film generate potential difference and breakdown. Through the thermal effect and electrochemical reaction (including plasma reaction and free radical reaction) generated on the surface of SiC single crystal, the thinning layer is formed on the surface of SiC, and then the film is easily removed by mechanical method, and finally the polishing effect is achieved. Because the discharge stage in the PEECMP method is in the glow discharge stage, the discharge energy is small, therefore, the SiC surface produced a thin layer which can be easily peeled off, and the electrolyte used in PEECMP are neutral salt solution, PEECMP is an efficient and environmentally friendly polishing method.

The PEECMP process is very complex and the mechanism is still unclear. Power supply voltage, discharge frequency and duty ratio are important parameters in the process of PEECMP, so it is necessary to study the influence of each factor on the polishing efficiency and workpiece surface roughness. In this paper, the influence of voltage, frequency and duty ratio on the final surface roughness (Ra) and polishing efficiency of plasma enhanced electrochemical mechanical polishing SiC were investigated.

2. Experimental section

The SiC single crystal used in this experiment is a 4 inch N-type 4H-SiC single wafer, which with a thickness of 460μm and a resistivity of 0.015~0.028Ω cm. All experiments are carried out on Si plane. The SiC is cut into 10 mm×10 mm single sample. The sample surface is wiped with acetone and alcohol, soaked with deionized water and dried with a dryer. Plasma enhanced electrochemical mechanical polishing experimental equipment is mainly composed of plasma discharge equipment and mechanical polishing equipment. The mechanical polishing equipment adopts PG-1 metallographic sample polishing machine, and the plasma discharge equipment mainly includes power supply, electrolyte system and the main part of the machine tool (as shown in Figure 1). JCL-DSP3 high frequency unipolar pulse power supply was used in this experiment. The voltage, frequency and duty ratio of this power supply are independently adjustable. The positive electrode of the power supply is connected to the SiC as the anode, and the negative electrode of the power supply is directly connected to the electrolytic cell made of stainless steel as the cathode. The size of the electrolytic cell is 300mm ×500mm×200mm, and NaCl with the concentration of 10g/L is used as the electrolyte. The mechanical polishing was carried out with a concentration of 30% silicon dioxide, whose diameter is 5μm.

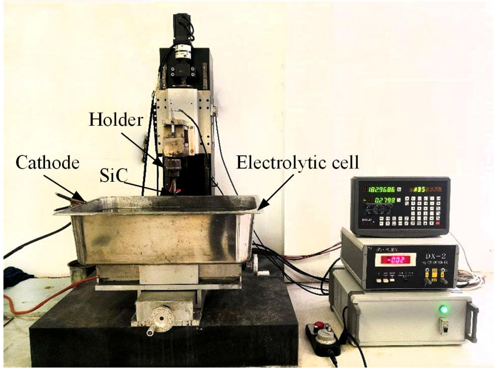

Figure 1. Plasma discharge experimental system.

The scanning white light interferometer (SWLI) is used to measure the roughness of SiC surface. By measuring the thickness of SiC before and after polishing with micrometer, the plasma enhanced electrochemical mechanical polishing efficiency of SiC was calculated.

3. Results and Discussion

3.1 The influence of voltage on polishing efficiency and surface roughness

In the process of plasma enhanced electrochemical mechanical polishing of SiC, the influence of designed peak voltage on polishing efficiency and SiC surface roughness of the experimental parameters are shown in Table 1.The power supply voltages in this experiment were selected as 80V, 90V, 100V, 110 and 120V. The polishing efficiency was calculated by measuring the thickness difference of SiC before and after machining, and the roughness was measured by white light interferometer. In order to reduce experimental error, each group of experiments was conducted three times, and the average value of the three experiments was taken as the final experimental result. Figure 2 is the relationship between peak voltage and process results.

Table 1. Experimental parameters at different voltages.

Experimental Parameters	Values
Voltage（V）	80、90、100、110、120
Frequency（kHz）	60
Duty Ratio	50%
Time	20min

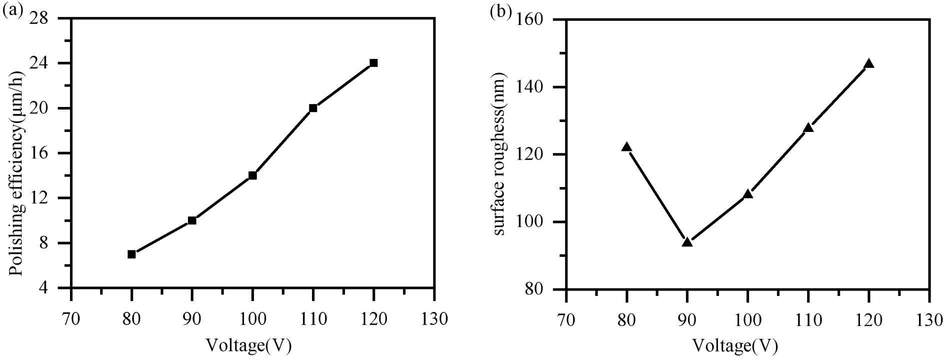

Figure 2. Effect of peak voltage on polishing efficiency and surface roughness.

Figure 2 (a) shows the trend of polishing efficiency changing with the power supply peak voltage. It can be seen from the figure, polishing efficiency increases rapidly with the continuous increase of peak voltage. This is because the total input electric energy of the discharge system increases, the electrochemical reaction speed is accelerated, and a large number of oxygen plasma and strong oxidizing free radicals are generated to oxidize the silicon carbide surface into oxide film layer, and the oxide film thickness increases, thus increasing the polishing efficiency.

Figure 2 (b) shows the variation trend of SiC surface roughness with the peak voltage of power supply. It can be seen from the figure that the SiC surface roughness decreases then increases with the increase of the peak voltage of power supply. Because the hardness of abrasive particles used in the mechanical polishing process is much smaller than silicon carbide, therefore, the mechanical polishing process is only to remove the oxide film layer on the surface of SiC, that is, the final surface roughness of silicon carbide is determined by the interface between the oxide film layer and SiC. When the oxide film is very thin, the high speed diffusion of hydroxyl free radicals through the oxide film, makes it difficult to achieve uniform diffusion of the entire workpiece surface, so the interface between the oxide film and SiC is very rough. When the oxide film gradually thicken, the densification of the oxide film becomes better, and the interface between the oxide film and SiC becomes flatter[10]. Therefore, in the initial stage of power supply peak voltage rise, the oxide film thickness increases with per unit time, and the SiC surface roughness decreases. When peak voltage continues to rise, it can be observed the electrochemical reaction becomes more severe, the cavitation force produced by the oxygen bubbles breaking makes the SiC surface part of the oxide film removed, so as to make the thickness of the remaining oxide film on the SiC surface decreased, and the new hydroxyl radicals diffusion through the oxide film layer with high speed, SiC surface roughness is increased.

3.2 The influence of power frequency on polishing efficiency and surface roughness

The experimental parameters about the influence of power frequency on polishing efficiency and surface roughness are shown in Table 2. Under the condition that the other parameters such as peak voltage and duty ratio of power supply unchanged, plasma enhanced electrochemical mechanical polishing experiments of SiC were carried out with power frequency of 20 kHz, 40 kHz, 60 kHz, 80 kHz and 100 kHz respectively. The polishing efficiency was calculated and the surface roughness was measured. Similarly, each group of experiments was carried out for three times, and the average value of the three experiments was taken as the final experimental result. Figure 3 is the relationship between the power discharge frequency and the results of each process.

Table 2. Experimental parameters at power frequency.

Experimental Parameters	Values
Peak Voltage（V）	100
Frequency（kHz）	20、40、60、80、100
Duty Ratio	50%
Time	20min

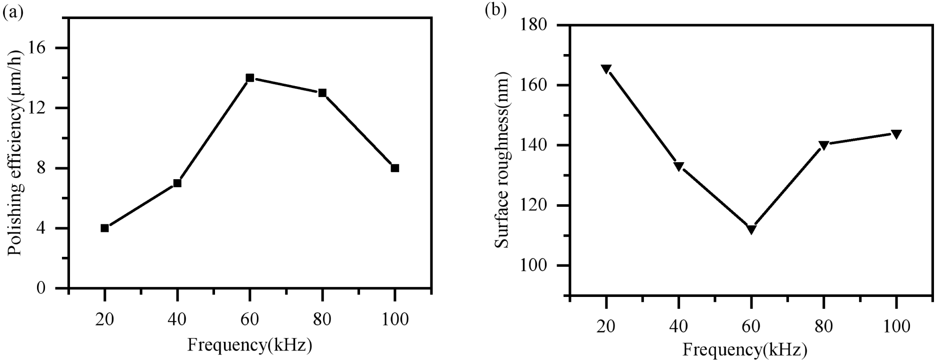

Figure 3. Effect of power frequency on polishing efficiency and surface roughness.

Figure 3 (a) shows the trend of SiC polishing efficiency changing with power discharge frequency. It can be seen from the picture, with the increase of discharge frequency, the polishing efficiency increases quickly. This is because with the increase of discharge frequency, gas ionization rate increased, speeds up the oxygen plasma and some strong oxidizing free radicals produce rate, so that the SiC surface oxidation rate is improved, the oxidation film thickness is increased, then in the mechanical polishing process, the oxidation film stripping ratio is increased. However, as the power supply frequency continues to rise, when the duty ratio is constant, the power supply pulse width decreases, the oxide film thickness of per unit time decreases. The relationship between pulse width and power supply frequency and duty ratio is shown as follows:

$$T_{on} = \frac{1}{f} \times D_{on} \tag{1}$$

Figure 3 (b) shows the trend of SiC surface roughness changing with power discharge frequency. It can be seen from the figure that with the increase of discharge frequency, the generation rate of oxygen plasma and strong oxidizing free radical increases, the thickness of oxide film increases and the density is good, the interface between SiC and oxide film gradually becomes gentle, and the surface roughness after mechanical polishing decreases. With the continuous increase of discharge frequency, when the duty ratio is constant, pulse width decreases, the thickness of oxide film produced per unit time decreases, the density is not good enough, and the surface roughness after mechanical polishing increases.

3.3 The influence of duty ratio on polishing efficiency and surface roughness

The parameter to explore the influence of power duty ratio on polishing efficiency and surface roughness is shown in Table 3. The experiments on polishing efficiency and surface roughness were also carried out for three times, and the average value of the three experiments was taken as the final experimental result. Figure 4 shows the relationship between power duty ratio and each process result.

Table 3. Experimental parameters at different duty ratio.

Experimental Parameters	Values
Peak Voltage（V）	100
Frequency（kHz）	60
Duty Ratio	10%、30%、50%、70%、90%
Time	20min

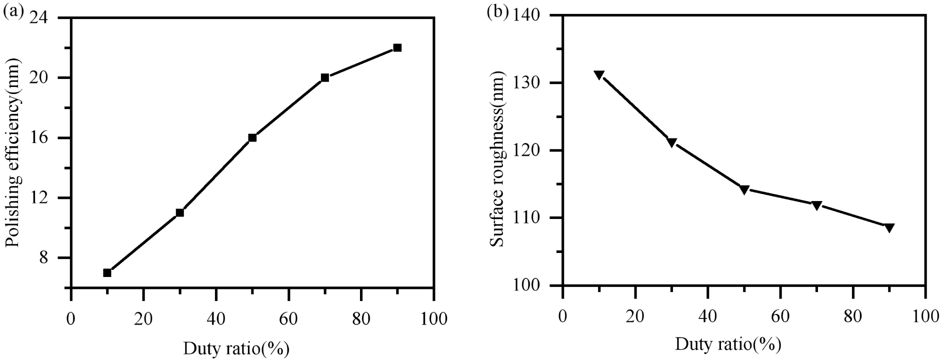

Figure 1. Effect of duty cycle on polishing efficiency and surface roughness.

The relationship between duty ratio and SiC polishing efficiency is shown in Figure 4 (a). It can be seen from the figure that the polishing efficiency of SiC increases gradually with the increase of duty ratio. The reason for this phenomenon is that when the power discharge frequency is constant, with the increase of duty ratio, the width of single pulse increases, that is, the discharge time within a single pulse increases, the energy utilization rate in each unit time increases, the oxide film thickness on the SiC surface increases, and the polishing efficiency becomes higher. But as the duty ratio continues to increase, the oxide film thickness increases rapidly and tends to be stable, and the polishing efficiency tends to be stable.

Figure 4 (b) shows the relationship between duty ratio and SiC surface roughness. It can be seen that, with the increase of duty ratio, the pulse width increases, a single cycle of discharge time becomes longer, the thickness of SiC surface oxidation film increases. The oxygen plasma and strong oxidizing free radicals is difficult to pass through the oxide film layer, oxide film and SiC interface become more flat. The SiC surface roughness is decreased by mechanical polishing. And as the power duty ratio increases, the oxide film thickness gradually tends to be stable, so the surface roughness becomes gentle. Please keep a second copy of your manuscript in your office. When receiving the paper, we assume that the corresponding authors grant us the copyright to use the paper for the book or journal in question. Should authors use tables or figures from other Publications, they must ask the corresponding publishers to grant them the right to publish this material in their paper.

4. Conclusion

In this paper, the influence of voltage, frequency and duty ratio on the surface roughness and polishing efficiency of plasma-enhanced electrochemical mechanical polishing for SiC single crystal were investigated. The results are as follows:

(1) With the increase of voltage, polishing efficiency becomes higher, and SiC surface roughness increased, then decreased.

(2) When the power frequency increases, the surface roughness decreases, and the polishing efficiency first increases and then decreases.

(3) With the increase of duty ratio, polishing efficiency increased and surface roughness decreased.

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