

High-integrity finishing of 4H-SiC (0001) by plasma-assisted polishing

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Abstract. A novel machining method combined with the irradiation of atmospheric pressure plasma was proposed for the finishing of difficult-to-machine materials. The irradiation of helium-based water vapor plasma efficiently oxidized the surface of single-crystal 4H-SiC (0001), and a ball-on-disc test using an alumina ceramic ball revealed that the wear rate of SiC, the surface of which was modified by the irradiation of water vapor plasma, is 20-fold higher than that of the surface without plasma irradiation. Plasma-assisted polishing using CeO₂ abrasives enabled us to improve the surface roughness of SiC without introducing crystallographical subsurface damage, and a scratch-free surface with a roughness of less than 0.3 nm rms was obtained.

Introduction

Silicon carbide (SiC) is a promising next-generation semiconductor power device material because it has excellent properties, such as a wide energy band gap, a high thermal conductivity, high saturated electron drift velocities and high-breakdown electric fields. In the finishing of the SiC substrate for an electronic device, chemical mechanical polishing (CMP) with diamond abrasives is generally performed. The polishing rate of SiC in CMP is low (less than 0.5 $\mu\text{m}/\text{h}$), and microscratches and subsurface damage, which deteriorate the surface integrity, are inevitably generated on the surface. Therefore, it is generally difficult to obtain an adequate level of surface integrity for epitaxial growth in a commercially available SiC wafer because of its high hardness and chemical inertness [1, 2].

Recently, some flattening techniques based on new concepts, which use Pt as a catalyst in HF solution [3,4] or the irradiation of ultraviolet light [5], have been developed. In these techniques, a scratch-free surface without introducing subsurface damage is obtained. On the other hand, we proposed a novel finishing technique, which combines the irradiation of atmospheric pressure plasma for surface modification, to realize the high-efficiency and high-integrity finishing of difficult-to-machine materials.

In this paper, we describe the machining properties of the newly proposed plasma-assisted lapping technique, such as surface roughness and subsurface damage, for 4H-SiC substrates.

Experimental setup

Fig. 1 shows the schematic of the experimental apparatus used. This apparatus consists of plasma generation and mechanical removal parts, which are separately installed to investigate the basic removal mechanism. Atmospheric pressure plasma is generated by applying a high-frequency ($f=13.56$ MHz) electric power, and helium containing water vapor (1.7-2.6%) or containing O₂ (5%) is supplied as a process gas with a flow rate of 1.5 L/min. Water vapor is introduced into the process gas by the helium bubbling of ultrapure water, and its concentration is measured using a dew-point meter. The copper electrode used is covered with a quartz glass to prevent arc discharge by generating dielectric barrier discharge. In the mechanical removal part, a ball-on-disc test with an alumina ball ($\phi 6.35$ mm) or a lapping /polishing test with a polishing film ($\phi 8$ mm) can be conducted by changing the tool head. The test sample is installed on the rotary table, and the surface modification by plasma irradiation and mechanical removal using an alumina ball or a lapping film are sequentially conducted. We use a 4H-SiC (0001) substrate as a specimen.

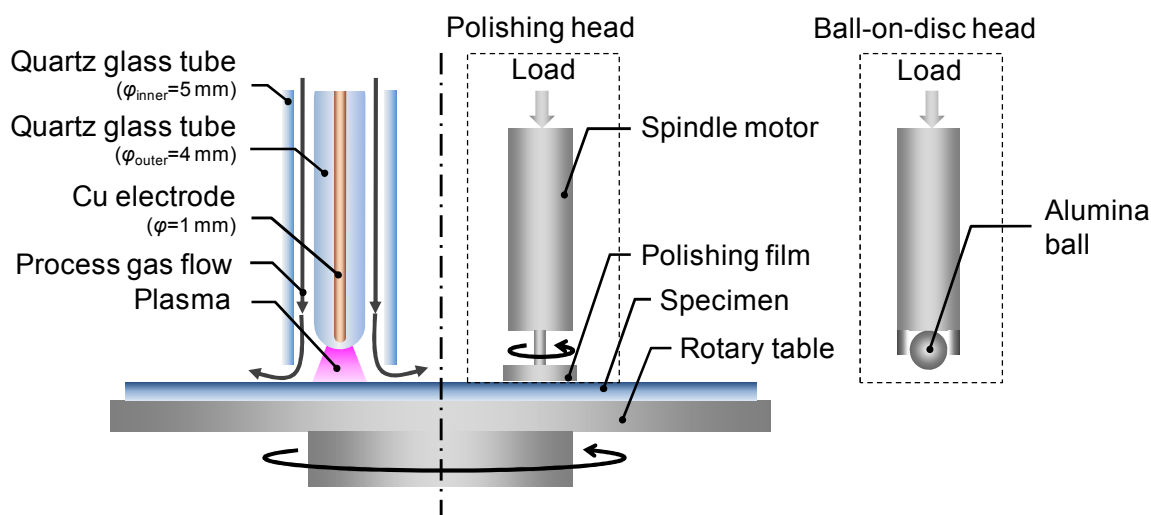


Fig. 1. Schematic of experimental apparatus.

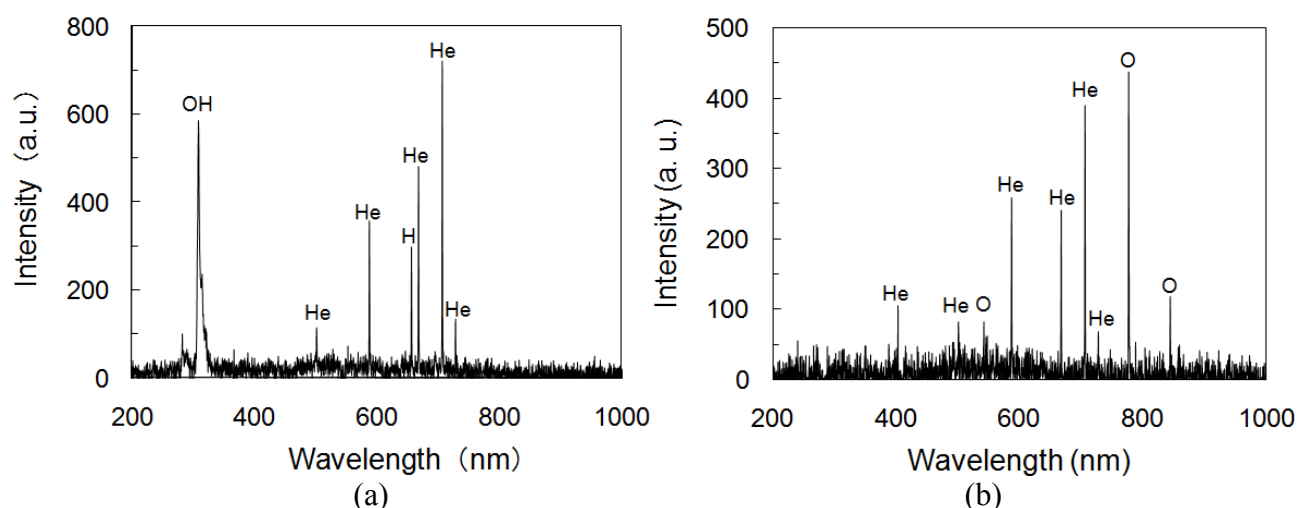


Fig. 2. Emission spectra of the plasma: (a) water vapor plasma, (b) oxygen plasma.

Results and discussion

Fig. 2 shows the emission spectra of the atmospheric pressure plasma. The emission spectra from the plasma were measured using a multichannel spectrometer (Ocean optics, USB4000). In the case of the water vapor plasma, strong emissions from OH molecules ($\lambda=309$ nm) were observed, as shown in Fig. 2(a), and in the case of the oxygen-containing plasma, emissions from oxygen atoms were observed, as shown in Fig. 2(b). From these measurement results, the major reactive species for surface modification in the water vapor and oxygen plasmas were considered to be OH and O radicals, respectively.

Table 1. Experimental parameters in ball-on-disc test

Ball material	Alumina
Load (g)	100
Rotating speed (mm/s)	69.1
Process gas	He + 2.6% H ₂ O, He + 5.0% O ₂
Flow rate (L/min)	1.5
RF power (W)	30
Processing time (min)	30

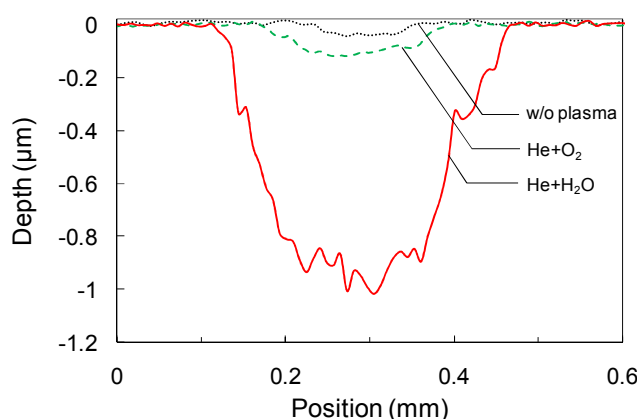


Fig. 3. Cross-section of SiC surface after ball-on-disc test.

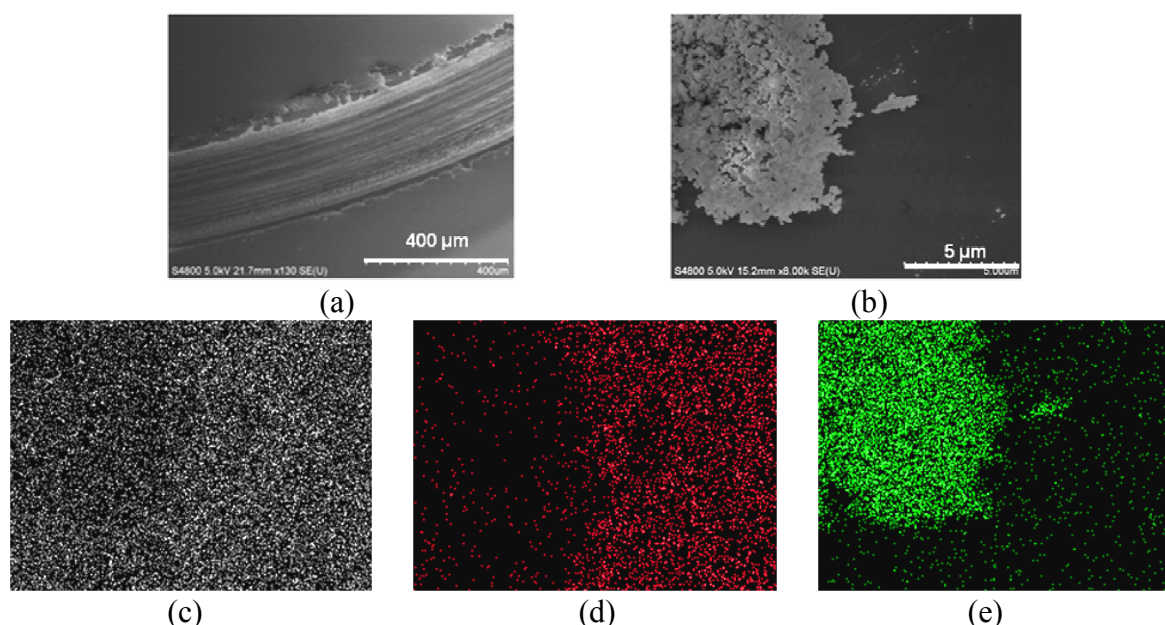


Fig. 4. SEM images of SiC surface after ball-on-disc test: (a) wear track, (b) close-up image of wear debris, element distribution images (c) silicon, (d) carbon, (e) oxygen.

Fig. 3 shows the cross sections of the SiC surface subjected to the ball-on-disc test, and the experimental parameters are shown in Table 1. These results indicate that the irradiation of the plasma promotes the increase in the depth of abrasion. In particular, the irradiation of the water vapor plasma increases the depth of abrasion more than 20-fold in comparison with the case without plasma irradiation. Fig. 4 (a) shows the SEM image of the SiC surface after performing the ball-on-disc test with water vapor plasma irradiation, and numerous pieces of wear debris are observed around the wear track. Figs. 4(c), (d) and (e) indicate that the element distribution images of the debris shown in Fig. 4(b) analyzed by energy-dispersive X-ray fluorescence spectroscopy (EDX) show the distributions of Si, C and O, respectively. From these images, it was presumed that the wear debris is silicon oxide because the quantity of Si did not change, whereas that of C decreased and that of O increased.

Fig. 5(a) shows the load-displacement curves of the SiC surface measured by the nanoindentation method. The Berkovich-type indenter made of diamond was used, and the maximum load was 0.5 mN. The hardness of the surface was calculated by the Oliver-Pharr method [6]. The following two types of specimen were evaluated: an unprocessed substrate and a substrate modified by the irradiation of the water vapor plasma to form an oxidized film with a thickness of about 100 nm. The obtained results shown in Fig. 5(b) indicate that the hardnesses of the unprocessed and modified surfaces are 37.4 GPa and 4.5 GPa, respectively. Hence, the irradiation of the water vapor plasma is very effective for softening the SiC surface.

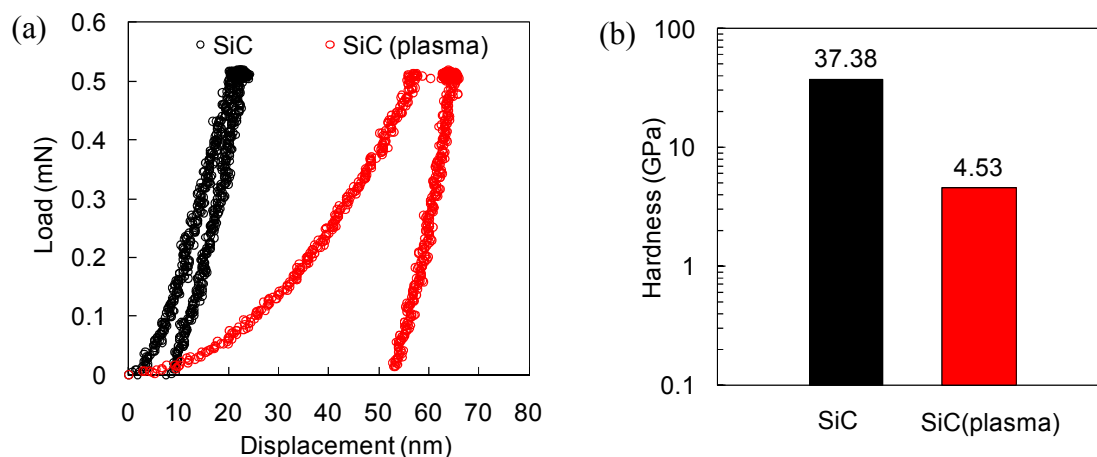


Fig. 5. Results of nano-indentation test of SiC: (a) load-displacement curve, (b) hardness calculated from measured data (a).

These results lead to the conclusion that the irradiation of the water vapor or oxygen plasma enables the removal of a SiC surface with a soft abrasive material by oxidizing the surface. Furthermore, the results of the ball-on-disc test imply that the oxidation power of OH radicals is higher than that of O radicals. This speculation is reasonable because the oxidation potential of OH (2.80 V) is greater than that of O (2.42 V).

Plasma-assisted polishing for the 4H-SiC (0001) 8° off-axis was conducted using a polishing film. The experimental parameters are listed in Table 2. The hardness of CeO_2 used as an abrasive in this experiment was smaller than that of SiC, but it was greater than that of SiO_2 . Therefore, it is expected that CeO_2 abrasives will remove only the oxidized layer without introducing scratches to the SiC surface. Fig. 6 shows the SEM image of the polishing film used in the experiment. The CeO_2 abrasives with a mean diameter of 0.5 μm were laminated on the PET film using a binder. Figs. 7(a) and (b) show the surface roughness images obtained before and after plasma-assisted polishing, which were measured using a microscopic interferometer (Zygo NewView 200CHR). No scratch was observed on the polished surface, while many scratches, which seemed to be introduced by diamond abrasives, were observed on the unprocessed surface, and a smooth surface with a roughness of less than 0.3 nm rms was obtained.

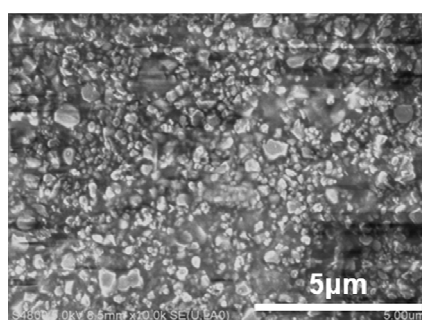


Fig. 6. SEM image of polishing film with CeO_2 abrasive.

Table 2. Experimental parameters in plasma-assisted-lapping

Abrasive	CeO_2 ($\phi 0.5\mu\text{m}$)
Load (g)	30
Rotating speed	69.1 mm/s (worktable), 5.2 m/s (lapping-spindle)
Process gas	He + 1.7% H_2O
Flow rate (L/min)	1.5
RF power (W)	6
Processing time (min)	60

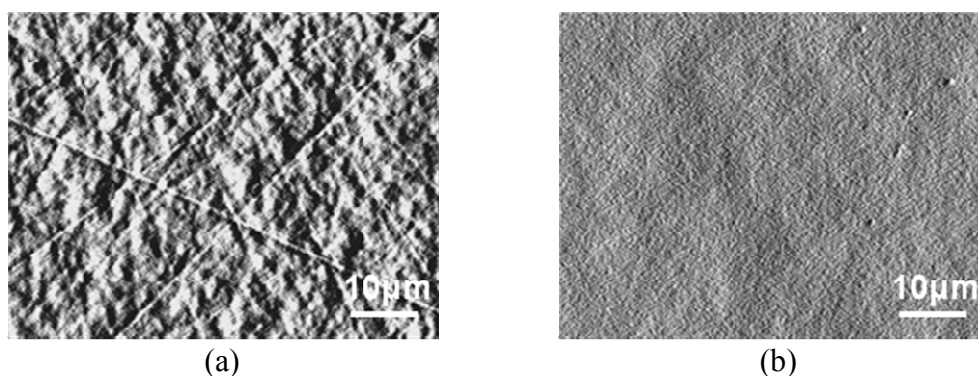


Fig. 7. Microscopic interferometer images of 4H-SiC (0001) surface: (a) unprocessed surface 4.410 nm p-v, 0.621 nm rms, (b) surface after plasma-assisted-lapping 1.889 nm p-v, 0.280 nm rms.

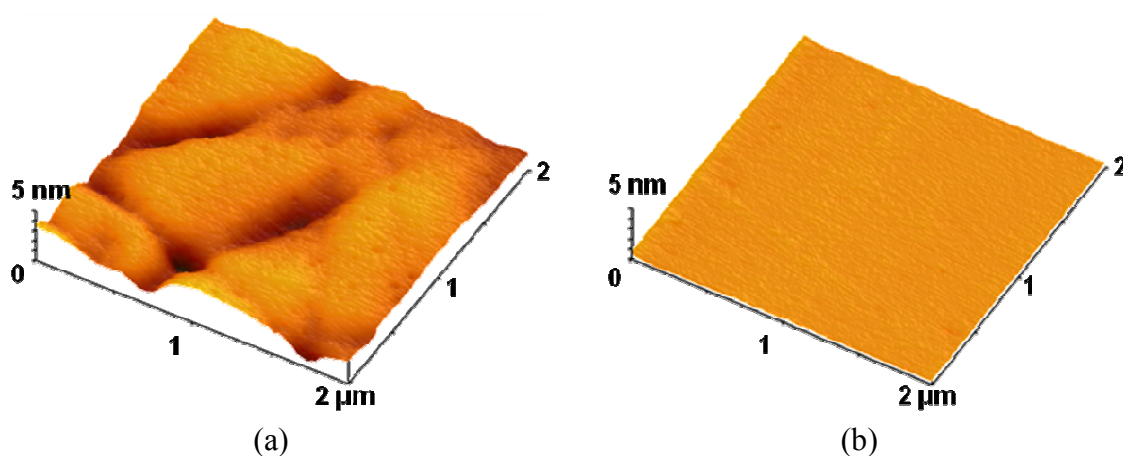


Fig. 8. AFM images of 4H-SiC (0001) surface: (a) unprocessed surface 3.648 nm p-v, 0.495 nm rms, (b) surface after plasma-assisted-lapping 1.319 nm p-v, 0.060 nm rms.

Figs. 8(a) and (b) show the AFM images of the unprocessed and processed surfaces. These images indicate that the surface roughness of SiC was improved in the spatial wavelength range of the micrometric level, and a very atomically smooth surface was obtained. The residual strains of the surface observed before and after polishing were evaluated by reflection high-energy electron diffraction (RHEED) measurement. RHEED measurements revealed that the lattice constants of the surface determined before and after plasma-assisted polishing are 0.316 nm and 0.308 nm, respectively. The lattice constant of an ideal SiC crystal is 0.307 nm. These results lead us to conclude that the newly proposed plasma-assisted polishing technique enables us to obtain an atomically smooth surface in finishing a SiC substrate without introducing crystallographical subsurface damage.

Summary

A novel lapping/polishing technique combined with the irradiation of atmospheric pressure plasma was proposed for finishing difficult-to-machine materials. The results of EDX and nanoindentation measurements revealed that the irradiation of water vapor or oxygen plasma markedly decreases the hardness of SiC by oxidizing the surface, and the results of a ball-on-disc test indicated that the irradiation of water vapor plasma is superior to that of oxygen plasma for increasing the removal rate of SiC. The plasma-assisted polishing of SiC using CeO₂ abrasives removed scratches and crystallographical damage, which were observed on the surface of an unprocessed substrate. Therefore, plasma-assisted polishing is a very feasible way of obtaining an atomically smooth surface in finishing a SiC substrate without introducing subsurface damage.

Acknowledgments

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