Electrochemical mechanical polishing of 4H-SiC (0001) with different grinding stones

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Keywords: SiC, electrochemical mechanical polishing, grinding stone, polishing property.

Abstract. Single-crystal SiC (4H-SiC) is a promising next-generation semiconductor power-device material because of its excellent electronic and thermal properties, such as a wide band gap, high breakdown field and high thermal conductivity. As a means of realizing the high-quality, highly efficient and low-cost polishing of SiC, electrochemical mechanical polishing (ECMP) was proposed. In ECMP, the hard SiC surface is modified to a soft oxide layer by anodic oxidation then the soft oxide layer is removed by abrasives softer than SiC. In this study, three types of grinding stones, alumina, silica and ceria, were used to polish the Si face of a 4H-SiC substrate in the ECMP process, and the polishing properties of the different grinding stones were evaluated in terms of the surface roughness and material removal rate (MRR) of the processed SiC surface.

Introduction

Single-crystal SiC (4H-SiC) has many excellent thermal and electronic properties, such as a wide band gap, high breakdown field, high thermal conductivity, and so forth. Therefore, it is one of the most promising materials for applications under extreme conditions. To realize applications utilizing SiC, smooth surfaces without scratches and subsurface damage (SSD) are essential [1]. However, SiC is difficult to machine owing to its high hardness and chemical inertness. Various polishing techniques, such as plasma-assisted polishing (PAP) [2], catalyst-referred etching (CARE) [3] and chemical mechanical polishing (CMP) [4], have been developed to finish SiC substrates, and atomically smooth surfaces have been obtained using these techniques. However, the material removal rate (MRR) of these techniques is not sufficient for practical manufacturing.

To realize the highly efficient polishing of SiC with minimal SSD, several electrochemical mechanical polishing (ECMP) techniques have been proposed [5-7]. In ECMP, the hard SiC surface is modified to a soft oxide layer by anodic oxidation and then the soft oxide layer is removed by soft abrasives. We previously proposed ceria-slurry-based ECMP [6], in which ceria slurry was used as the electrolyte for anodic oxidation and as a polishing medium to remove the oxide layer, resulting in the efficient combination of anodic oxidation and abrasive polishing. Since anodic oxidation and polishing are simultaneously conducted, an MRR of 3.62 μm/h for a diamond-abrasive-polished SiC surface was obtained, and the decrease in the root-mean-square (RMS) roughness was very rapid; the RMS roughness decreased from 0.97 nm *Sq* to 0.23 nm *Sq* after 30 min of ECMP. Ceria-slurry-based ECMP is thus very efficient for the finishing of SiC surfaces.

Since the slurry was used as an electrolyte and a polishing medium, control of the oxidation and polishing conditions is difficult, and the type of slurry that can be used is limited. Therefore, here we propose a slurryless ECMP process, in which fixed soft abrasives instead of loose abrasives are used to remove the oxide layer. This enables a separate electrolyte to be used for the anodic oxidation and the performance of ECMP can be improved. In this study, experiments were conducted to select a suitable grinding stone for the ECMP of SiC. Three types of grinding stones, alumina, silica and ceria, were used to polish the Si face of a 4H-SiC substrate in the ECMP process, and the polishing properties of the different grinding stones were evaluated in terms of the surface roughness and MRR of the processed SiC surface.

Experiment

Fig. 1 shows a schematic of our proposed slurryless ECMP apparatus, which consists of an anodic oxidation unit and a polishing unit. The anodic oxidation unit consists of a three-electrode system: a work electrode (WE, SiC substrate), reference electrode (RE, Ag|AgCl) and counter electrode (CE, SUS plate). The anodic oxidation process is controlled by a potentiostat. The SiC substrate is fixed on a copper plate and serves as the WE. A grinding stone is fixed on the head of the spindle, and an elastic resin is set between the grinding stone and the spindle to automatically adjust the tilt between the grinding stone and the SiC surface and confirm uniform contact between them. The polishing load is provided by weights placed on the top of the spindle. A pump is used to recycle the electrolyte and prevent the electrolyte from being ejected from the polishing area owing to the centrifugal force of the spindle.

Commercially available single-crystal 4H-SiC substrates (3 inch, 4°-off, n-type) were employed in this study. Both faces of each substrate were prepared by two-step diamond lapping. Each face was first lapped with a #4000 diamond grinding plate (average particle size of 3 μm) for 10 min at a pressure of 19.6 kPa, then it was lapped with a #20000 diamond grinding plate (average particle size of 0.3 μm) for 30 min at the same pressure. ECMP was conducted on the Si face of the substrate. Table 1 shows the ECMP parameters; a sodium chloride (NaCl) aqueous solution with a concentration of 1 wt% and an electric conductivity of 1.8 S/m was used as the electrolyte and a current with a constant density of 0.2 mA/cm^2 was applied during the anodic oxidation. Polishing was conducted with a polishing pressure of 140 kPa and a spindle rotation speed of 500 rpm. Three types of vitrified bond grinding stones,

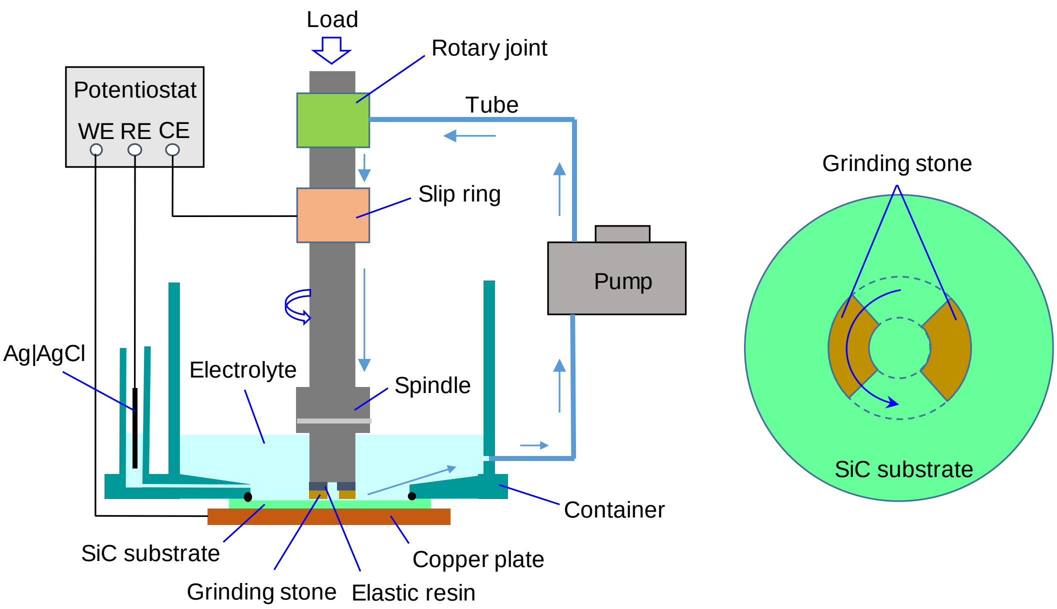

Fig. 1. Schematic of slurryless ECMP apparatus

Table 1. ECMP parameters

Parameters	Value
Current density [mA/cm^2]	0.2
Spindle rotation speed [rpm]	500
Polishing pressure [kPa]	140
Polishing time [min]	10
Electrolyte	1 wt% NaCl aqueous solution
Electrolyte flow rate [ml/min]	380
Vitrified bond grinding stone	Ceria, Silica, Alumina
Particle size	1 μm (#8000)

alumina, silica and ceria, with an average particle size of 1 μm (#8000), supplied by MIZUHO Co., Ltd., were applied. The processed surfaces were observed by atomic force microscopy (AFM, SPM 9700).

Results and discussion

Fig. 2 shows an AFM image of a SiC surface prepared by two-step diamond lapping that was used for ECMP. Although it had a low *Sa* roughness of 0.440 nm, many scratches were observed on the surface. These scratches were formed by diamond abrasives during the mechanical removal of the SiC surface. Since the hardness of diamond abrasives is higher than that of the SiC surface, the formation of scratches is inevitable in the diamond lapping of SiC.

Fig. 3 shows the morphologies of surfaces processed by ECMP with different grinding stones. Fig. 3(a) shows the anodic oxidized surface without simultaneous polishing; many oxide lines were generated on the surface. These oxide lines correspond to the scratches on the diamond-lapped surface since scratches were preferentially oxidized during the anodic oxidation of the SiC surface [8]. Fig. 3(b) shows the surface processed by ECMP with the ceria grinding stone; the scratches were removed and a smooth surface with an *Sa* roughness of 0.389 nm was obtained, almost the same as that of the initial diamond-lapped surface. In contrast, the surface roughness increased after ECMP with the silica and alumina grinding stones, as respectively shown in Fig. 3(c) and (d). From Fig. 3(c) and (d), it is clear that some of the oxide layer remained on the processed surface. This indicates that the MRR was lower than the anodic oxidation rate for the silica and alumina grinding stones. As the current density applied in the three experiments was the same, the oxidation rate was the same on the basis of Faraday's law of electrolysis. Thus, the results indicate that the ceria grinding stone had a higher polishing rate than the silica and alumina grinding stones during the removal of the oxide layer.

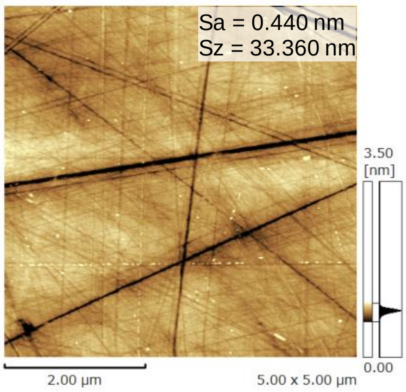

Fig. 2. Diamond-lapped surface.

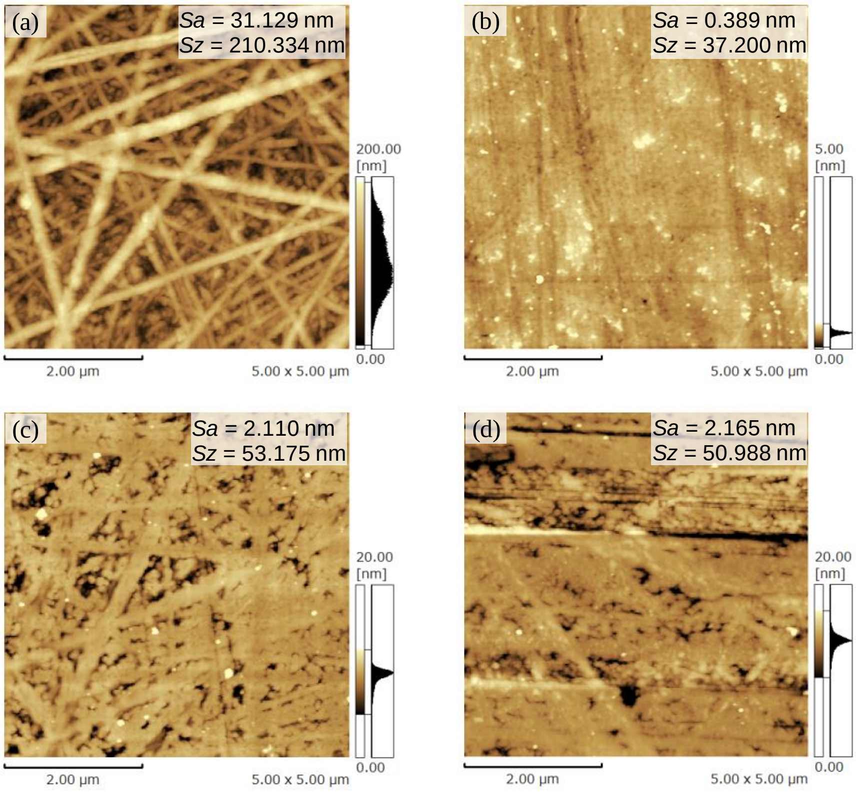

Fig. 3. Surface morphologies measured by AFM: (a) anodic oxidized surface without simultaneous polishing and surfaces processed by ECMP with (b) ceria grinding stone, (c) silica grinding stone and (d) alumina grinding stone.

To confirm the performance for the removal of the oxide layer for the three grinding stones, the depth of the polished area was measured by a stylus profiler as shown in Fig. 4(a). Fig. 4(b), (c) and (d) show cross-sectional views of the surfaces processed with ceria, silica and alumina grinding stones, respectively. Polishing depths of 0.25 μm, 0.12 μm and 0.15 μm relative to the oxidized area were obtained using the ceria, silica and alumina grinding stones, respectively. This shows that ceria is the most suitable material for removing the oxide layer. Since SiC was oxidized to silicon dioxide and silicon oxycarbides in the anodic oxidation process [9], the physical properties of the oxide layer are considered to be similar to those of quartz. Therefore, a quartz glass substrate was polished under the polishing conditions in Table 1 without electricity to verify the ECMP results.

Fig. 5 shows scanning white light interferometer (SWLI) images of quartz glass surfaces polished by the three types of grinding stones. Polishing groove with a depth of about 3.8 μm was observed on the surface processed with the ceria grinding stone as shown in Fig. 5(a). Compared with the ceria grinding stone, the application of the silica and alumina grinding stones resulted in lower MRRs with polishing depths of about 0.7 μm and 1 μm, as shown in Fig. 5(b) and (c), respectively. These results are consistent with the ECMP results. It has been found that ceria-based slurries are more capable of polishing silicon dioxide than silica slurries. This is because a chemical interaction occurs at the in ceria-silicon dioxide interface that enhances the polishing, although there is still a debate on the specific chemical interaction mechanism [10]. A chemical interactions also occurs in the polishing of silicon dioxide by silica-based slurries but it is much weaker than that for ceria-based slurries [11]. Therefore, it is assumed that the difference in the chemical interactions led to the higher MRR of the oxide layer for ceria than for silica and alumina, regardless of the relatively low hardness of ceria.

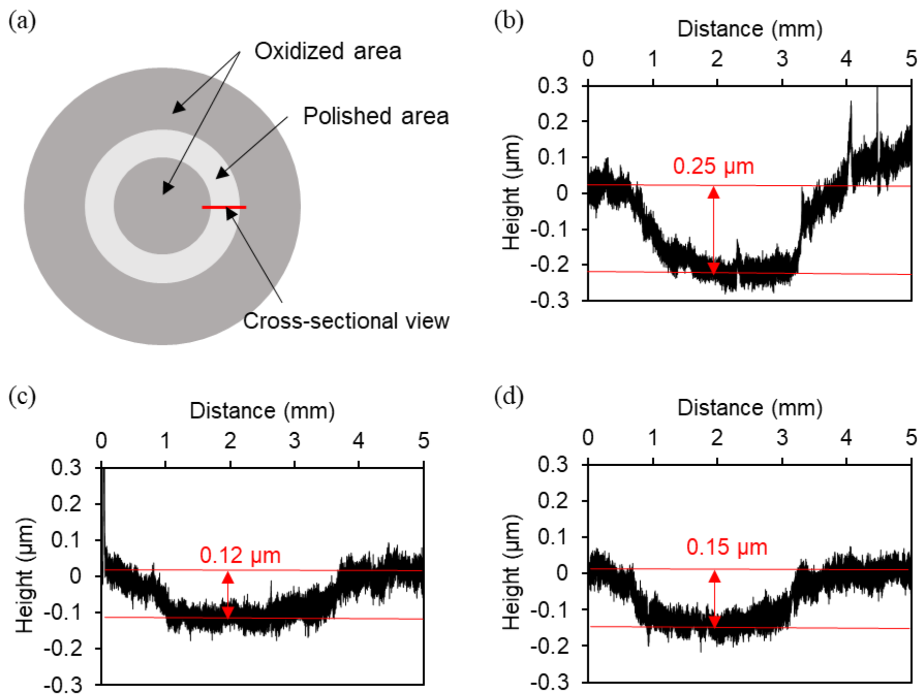

Fig. 4. Cross-sectional views of processed surfaces: (a) outline of the measurement and cross-sectional views of surfaces polished by (b) ceria grinding stone, (c) silica grinding stone and (d) alumina grinding stone.

The ECMP results shown in Fig. 3 also indicate that the balance between the oxidation rate and oxide polishing rate is very important in obtaining an atomically smooth surface; a smoother surface was obtained owing to the higher polishing rate of the ceria grinding stone than those of the silica and alumina grinding stones. However, a few areas of residual oxide existed on the ceria-processed surface, as shown in Fig. 3(b). This reveals that the oxide polishing rate was still lower than the anodic oxidation rate. It is expected that an atomically smooth surface can be obtained when the oxide polishing rate is greater than or equal to the anodic oxidation rate.

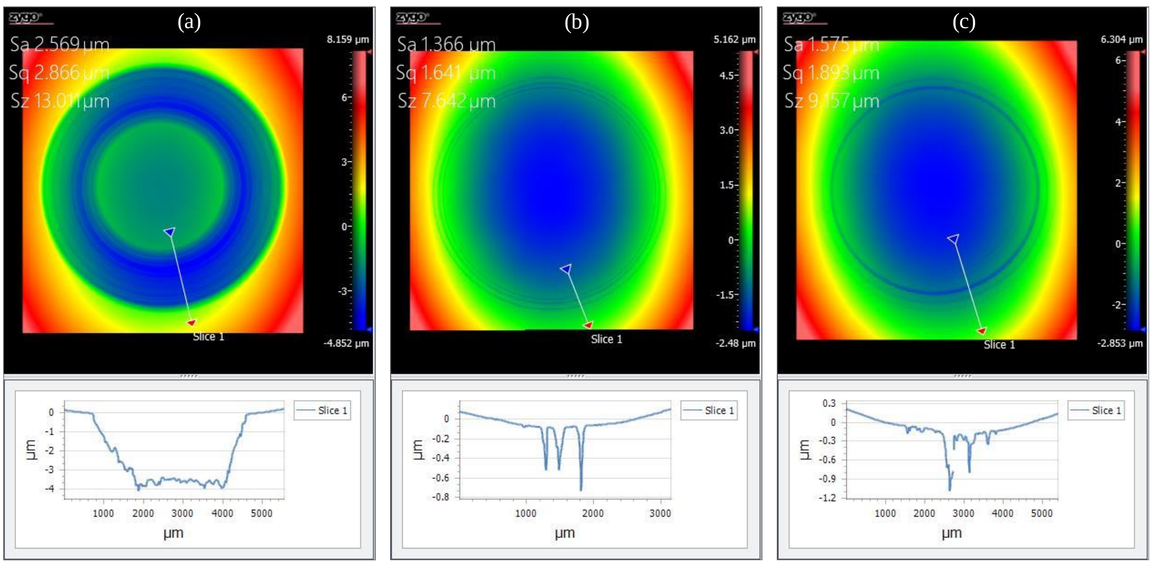

Fig. 5. SWLI images of polishing grooves on quartz glass surfaces processed by (a) ceria grinding stone, (b) silica grinding stone and (c) alumina grinding stone.

Conclusions

A 4H-SiC (0001) surface was processed by ECMP with three types of grinding stones, ceria, silica and alumina, and the polishing properties of the three grinding stones were evaluated. The ceria grinding stone had a better performance for the removal of the oxide layer than the silica and alumina grinding stones owing to its chemical interaction with silicon dioxide. A scratch-free surface was obtained by using the ceria grinding stone. To obtain an atomically smooth surface, the oxide polishing rate should be increased to the anodic oxidation rate.

Acknowledgements

This work was partially supported by a research grant from the Mitsutoyo Association for Science and a research grant from the Technology and Machine Tool Engineering Foundation.

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