Preparation of Ultrasmooth and Defect-Free 4H-SiC(0001) Surfaces by Elastic Emission Machining

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In this work, elastic emission machining (EEM), which is a precise surface-preparation technique using chemical reactions between the surfaces of work and fine powder particles, is applied to the flattening 4H-SiC (0001) surface. Prepared surfaces are observed and characterized by optical interferometry, atomic force microscopy (AFM), and low-energy electron diffraction (LEED). The obtained images show that the processed surface has atomic-level flatness, and the subsurface damage and surface scratches of the preprocessed surface are almost entirely removed.

Key words: Elastic emission machining (EEM), silicon carbide, surface morphology, atomic force microscopy (AFM), low-energy electron diffraction (LEED)

INTRODUCTION

Silicon carbide (SiC) is a promising semiconductor material for high-temperature, high-frequency, and high-power device applications because of its excellent properties, such as good thermal conductivity, good carrier mobility, and high chemical stability. For device applications, it is essential to prepare an atomically flat and defect-free SiC substrate because the initial surface condition of the substrate determines the quality of the epitaxial films used in device fabrication. Therefore, the surface preparation techniques have been extensively studied for achieving a well-ordered SiC substrate.

In general, surface-preparation techniques of the SiC surface can be classified into three types: polishing, chemical-mechanical polishing,[1,2] and chemical etching.[3–5] Polishing of SiC wafers uses hard abrasives, such as diamond polishing compounds mixed with water. The surface roughness and crystallographic damages can be reduced effectively with the use of diamond abrasives of submicron size. However, the material removal process is mechanical so that introduction of the damaged or strained subsurface layer containing cracks and/or dislocations is not avoided completely. On the contrary, chemical-mechanical-polishing methods combining mechanical polishing with a chemical etching action using silica or chromium III oxide powder particles has been reported to obtain defect-free surfaces. However, the process parameters, such as the condition of slurry and polishing pads, pressure, and temperature, are so difficult to control that damage-free and scratch-free surfaces cannot be obtained easily. The third one for smooth surface preparation is chemical etching employing hydrogen gas under atmospheric pressure. The hydrogen etching process at a temperature of approximately 1,500° C has recently been reported to obtain defect-free and scratch-free surfaces having a nearly vicinal structure. However, in order to flatten the large area on the SiC surface, the precise control of temperature and a large amount of thermal energy are required.

We have tried to prepare atomically flat and defect-free surfaces by elastic emission machining (EEM),[6–14] which uses the chemical reaction between the surfaces of the fine powder particles and the process surface. The EEM without a mechanical action does not generate defects, such as scratches and cracks, on the processed surface. The reactive species in EEM are atoms on the fine powder particles with a large diameter of more than 1 μm so that

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the atoms on the powder particles selectively interact with the topmost atoms on the processed surface. In terms of a Si(001) surface processed by EEM, 95% of the observed area (100 nm × 100 nm) was proven to be composed of only three atomic layers.[14] Therefore, EEM is expected to achieve an atomically flat and defect-free SiC surface. In this study, EEM is applied to flattening 4H-SiC (0001) surfaces, and we evaluate the flattening performance and crystallographic nature of the processed surface in comparison with a commercial SiC surface.

EXPERIMENTAL PROCEDURE

In EEM, powder particles are employed as a work solid that reacts with the target material. They are uniformly mixed with ultrapure water and are supplied to the process surface. When the powder particle surface and process surface come into contact with each other and separate, there is a notable probability that the atoms on one surface will adhere and move onto the other. In this study, a slit-type nozzle head was employed to transport the powder particles to the process surface. Figure 1 shows a schematic drawing of the slit-type nozzle head used in EEM, and Table I shows the process parameters. The distance between the nozzle and the process surface was set to 1 mm. So, the position of the nozzle is easy to control. The surface morphology and crystallographic structure of the processed SiC surface were evaluated by several surface-analytical techniques. An optical interferometry (ZYGO Corp., Middlefield, CT, NewView200CHR) and atomic force microscopy (AFM, SII Nanotechnology, Inc., Chiba, Japan, SPA400+SPI3800N) were used to evaluate the morphology of the processed and preprocessed surfaces. A silicon single-crystal cantilever of which the typical values of apex radius and spring constant were 5–10 nm and 40 N/m was employed in the AFM measurement. The crystallographic property of the processed surface was investigated by low-energy electron diffraction (LEED).

Table I. Specifications of Process Parameters Conditions

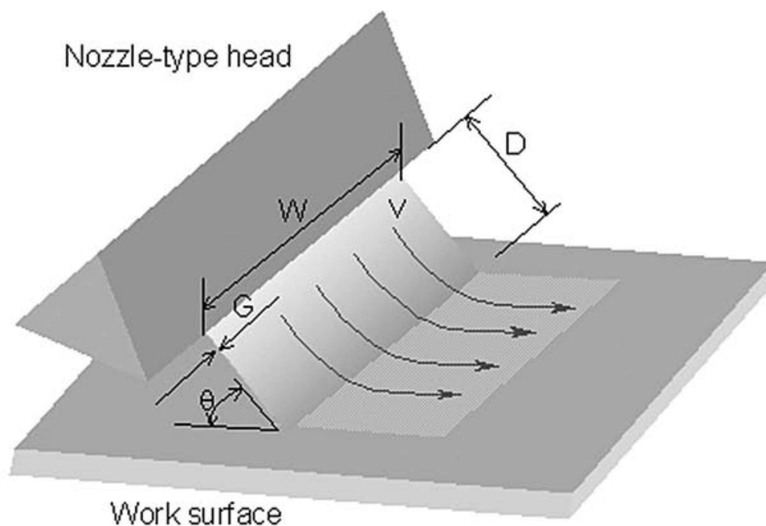
Workpiece	4H-SiC(0001)
Material of fine powder particles	SiO_2
Diameter of fine powder particle	1–10 μm
Volume concentration of fluid	6.8 vol.%
Temperature of fluid	23°

The removal depth of the processed surface was measured using an optical interferometer and was determined by the cross-sectional profiles. The wafers used in this study were on-axis 4H-SiC$(0001)_{Si}$ substrates having a 2-in.-diameter obtained from Nippon Steel Co., Ltd. (Tokyo). The wafers are n-type doped; their resistivities are 0.02–0.03 Ωcm; and the micropipe density was reported to be 50–100 cm^{-2} by the manufacturer. As a treatment before EEM, SiC substrates were dipped into a 50% HF solution for 10 min to remove the native oxide layer. The EEM-processed samples were treated with ultrapure water under irradiation with a 1-MHz supersonic wave for 5 min in order to remove the residual powder particles on the surface. Then, they were dipped into a 50% HF solution for 10 min and were rinsed with ultrapure water for 1–2 min. Silica powder particles (TOSOH SILICA Corp., Tokyo, Japan, NIPSIL 200A) were used as the reactive species of the SiC surface in this experiment.

RESULTS AND DISCUSSION

Optical Interferometer Observation

To investigate the flattening performance of EEM, processed surface roughness was measured using the optical interferometer. Figure 2a and b shows the slope image and the cross-sectional profile of the preprocessed surface, respectively. The observed area is 64 μm × 48 μm. Many scratches are observed on the preprocessed surface, and the surface mainly consists of wavelike protrusions having a spatial



Specifications of nozzle head process parameters

Gap of slit : G	300 μm
Aperture width of slit : W	5 mm
Incident angle : θ	35°
Facing distance : D	1 mm
Initial velocity of fluid : V	30 m/s

Fig. 1. Schematic drawing of nozzle-type head.

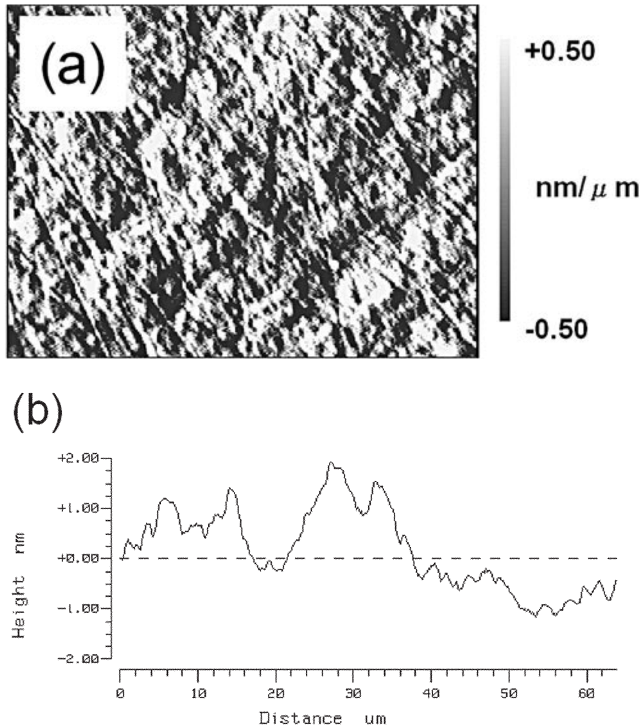


Fig. 2. Obtained image of preprocessed 4H-SiC(0001) surface: (a) slope profile and (b) typical cross section of surface. The microroughness of the preprocessed surface was Ra: 0.530 nm [P-V: 3.144 nm, RMS: 0.657 nm]. Measurement area is 64 μm × 48 μm.

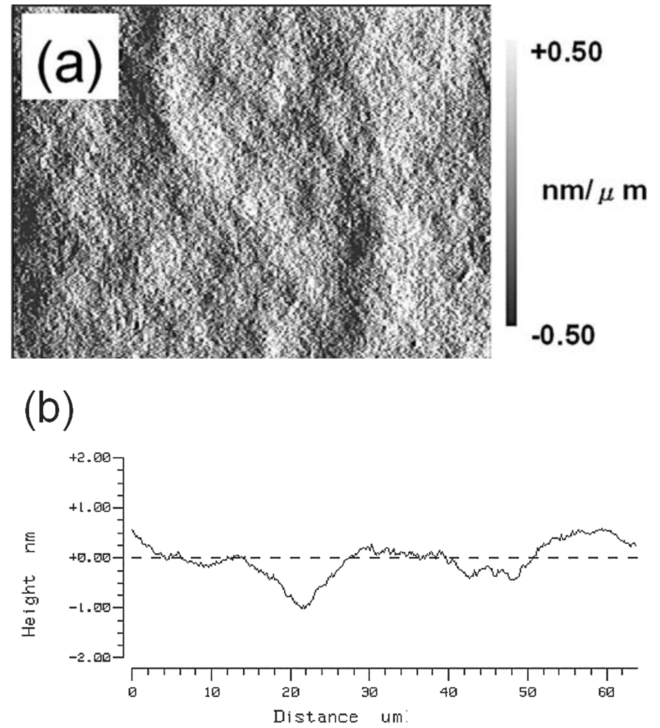


Fig. 3. Obtained image of processed 4H-SiC(0001) surface: (a) slope profile and (b) typical cross section of surface. The microroughness of the processed surface was Ra: 0.270 nm [P-V: 1.513 nm, RMS: 0.323 nm]. Measurement area is 64 μm × 48 μm.

wavelength range from 1 μm to 10 μm. Figure 3a shows the slope image of the EEM-processed surface, and Fig. 3b shows the cross-sectional profile of the EEM-processed surface. The removal depth during the process was 50 nm. From Fig. 3a and b, the scratches are seen to be removed, and the surface, which consists of wavelike protrusions having a spatial wavelength range from 5 μm to 10 μm, is found to be improved markedly. In order to characterize the surface microroughness in specific spatial wavelength, the SiC surfaces before and after EEM processing were evaluated through power spectral density (PSD) analysis. Figure 4 shows the PSD of the surface profile measured using an optical interferometer. These spectra show that the surface is smoothed in the spatial wavelength range from 1 μm to 10 μm during EEM.

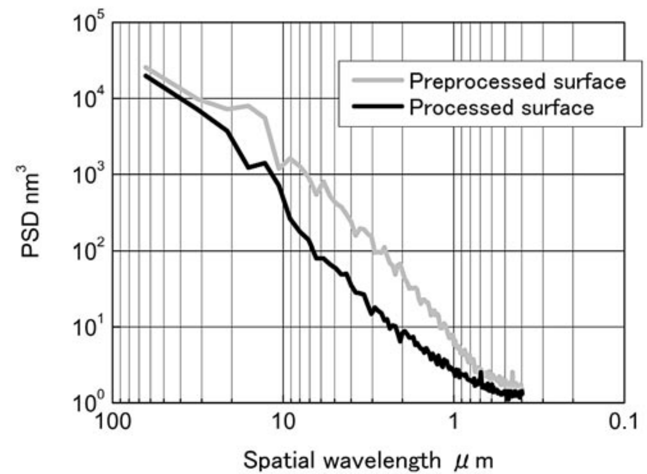


Fig. 4. The PSD spectra of preprocessed and processed surfaces.

AFM Observation

To evaluate the EEM-processed surface with a higher spatial resolution, the microroughness of the SiC surface was measured by AFM. Figure 5a shows an AFM image of 2 μm × 2 μm on the preprocessed surface after dipping into the 50% HF solution. The maximum height of irregularity (R_{pv}) and root-mean-square roughness (R_{rms}) are 5.477 nm and 0.696 nm, respectively. Scratches can be seen throughout the measurement area. Figure 5b shows an AFM image of the EEM-processed surface. The observed area is 2 μm × 2 μm. The microroughness of the surface is found to be 0.909 nm (R_{pv}) and 0.089 nm (R_{rms}). The surface morphology is completely different from the preprocessed surface shown in Fig. 5a. The scratches on the preprocessed surface are completely removed, and the roughness is markedly improved. The PSDs of the observed surface profiles were calculated and are shown in Fig. 6. The PSD analysis shows that the surface microroughness in the spatial wavelength range from 1 μm to 0.01 μm was improved by EEM.

LEED Observation

The crystallographic nature is an important factor determining the quality of the processed surface.

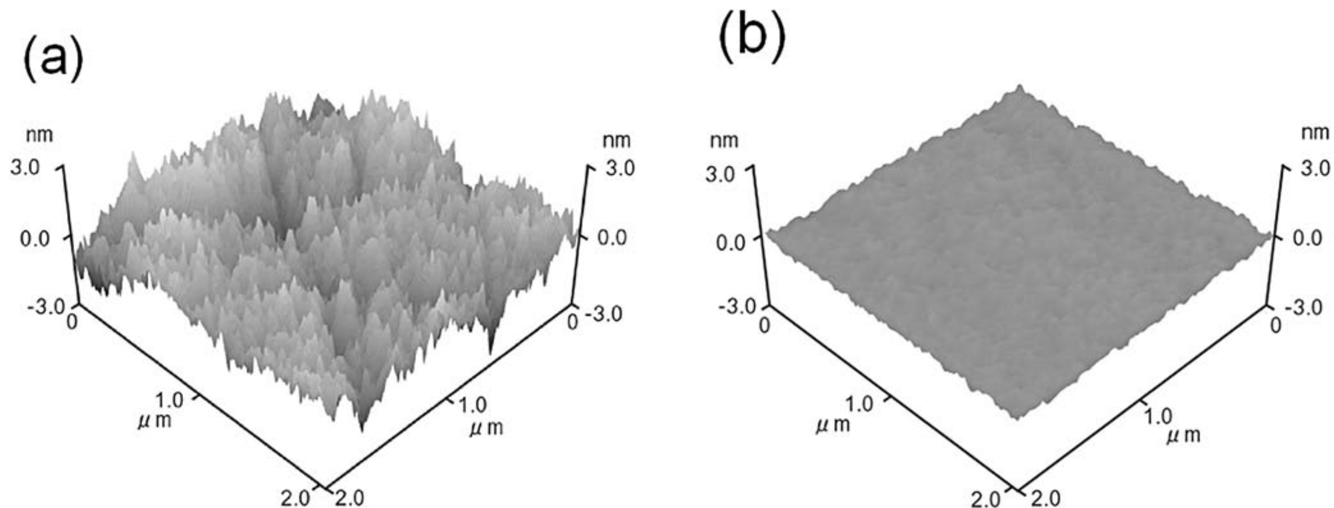


Fig. 5. The 2 μm × 2 μm AFM images of (a) preprocessed 4H-SiC(0001) surface and (b) EEM-processed 4H-SiC(0001) surface.

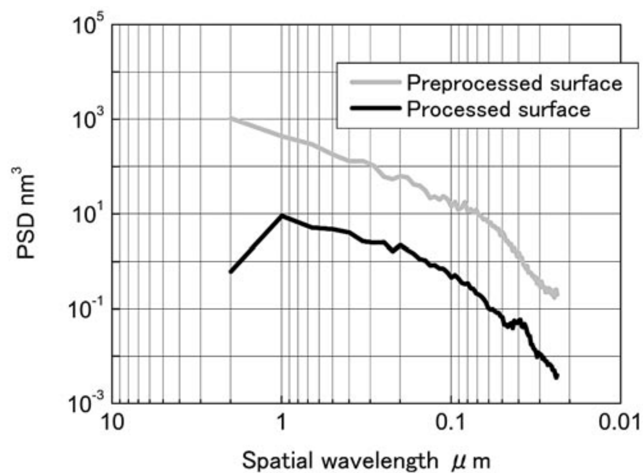


Fig. 6. The PSD spectra of profiles obtained by AFM.

Information about the atomic arrangement on the surface can be obtained from the LEED pattern. The preprocessed and EEM-processed surfaces were observed by LEED. Figure 7a shows the LEED pattern of the preprocessed surface after dipping into the 50% HF solution to remove the oxide layer. The electron beam energy is 85 eV and is sufficiently low to evaluate the damage caused during the surface preparation. The spot pattern could not be observed in the SiC surface after dipping into the 50% HF solution. This indicates that polishing-induced damage, the thickness of which is at least 1 or 2 atomic layers, exists on the SiC surface after dipping into the 50% HF solution. Figure 7b shows the LEED pattern of the EEM-processed surface. The LEED pattern of the processed surface clearly shows a diffraction spot of 1 × 1 caused by the crystal structure

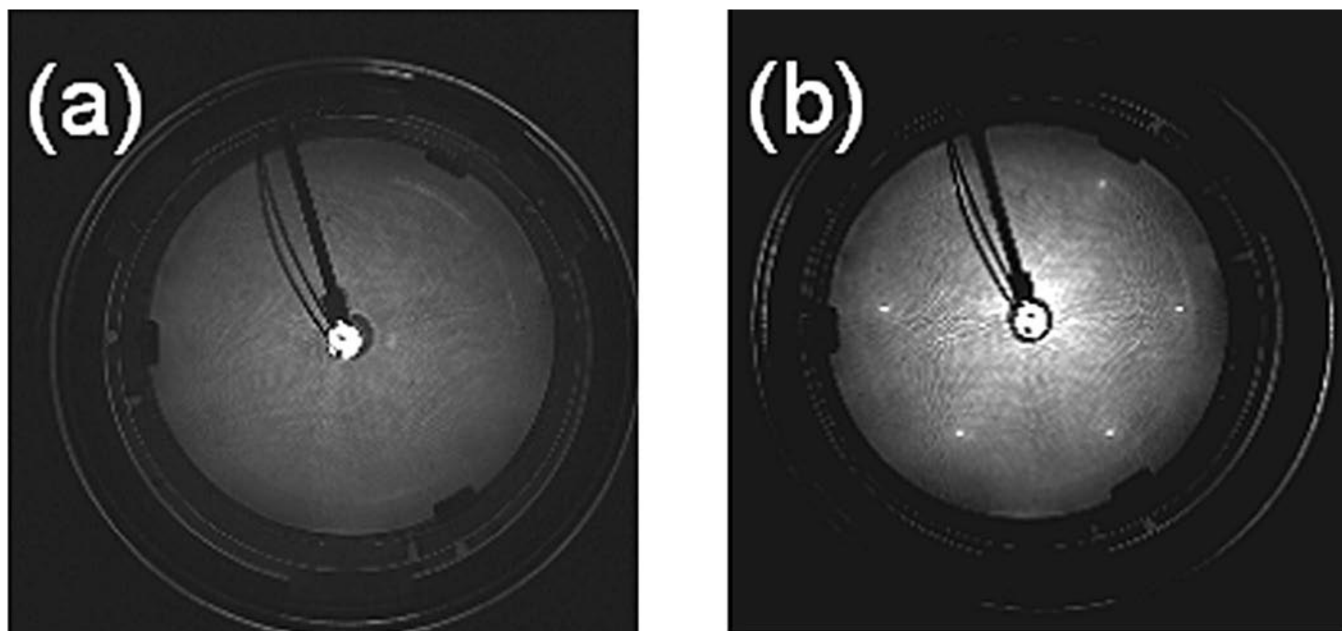


Fig. 7. Photographs of diffraction spots of (a) HF-cleaned 4H-SiC(0001) surface and (b) processed 4H-SiC(0001) surface. Electron energy is 85

of the bulk in contrast with that of the preprocessed surface. This result shows that the damage layer induced by the polishing during wafer production is removed by EEM and that the processed surface does not have a crystallographically disordered structure.

In this experiment, only the local area on the SiC surface was flattened by the nozzle with the aperture width of 5 mm. When the nozzle with the width of 2 in. is used, it can be estimated that a processing time of 12 h is required to remove a thickness of 15 nm on a full 2-in.-diameter wafer. This processing time is estimated when one nozzle works. However, many nozzles can actually be placed in parallel over the SiC surface, so the processing time can be shortened furthermore.

CONCLUSIONS

The (0001) surface of 4H-SiC, which is expected as a next-generation, power semiconductor device material, was flattened by EEM. We evaluated the flatness performance and crystallographic nature of the EEM-processed SiC surface. The processed surface has revealed that the innumerable scratches on the preprocessed surface could be completely removed by EEM. The microroughness of the processed surface was improved to be below 0.1 nm (R_{rms}) by EEM. The LEED observation of the processed surfaces indicates that not only a flat surface but also a crystallographically ordered surface could be prepared by EEM processing.

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