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Polishing-pad-free electrochemical mechanical polishing of single-crystalline SiC surfaces using polyurethane–CeO_2 core–shell particles

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ABSTRACT

A novel method for electrochemical mechanical polishing (ECMP) of the single-crystalline SiC surface, which has extremely high mechanical and chemical strength compared to conventional electronic materials, is reported. The method does not employ a polishing pad; it comprises electrochemical oxidization of the SiC surface and subsequent removal of the oxide by CeO_2 from the polyurethane–CeO_2 core–shell particles. The core–shell particles are used to maintain a gap between the polishing plate (cathode) and the SiC wafer (anode), which enables efficient anodic oxidation of the inert SiC surface. The core–shell particles, composed of the elastic polyurethane core covered with an abrasive layer of small and soft CeO_2 particles prepared by a simple and low-cost process, can be used to obtain a smooth SiC surface without using a polishing pad. The ratio of polyurethane to CeO_2 in the core–shell particles is optimized to obtain core particles that are fully covered with the shell particles without leaving excess CeO_2 particles. Using the fabricated core–shell particles, the conventional CMP process is unable to remove the SiC surface without anodization. While a continuous bias during polishing produces a rough SiC surface owing to the oxide film remaining on the treated surface, as confirmed by current measurements and X-ray analysis, a periodically applied bias, whose conditions were determined by the theoretical growth rate and residual thickness of the oxide film, reduces the number of scratches, and a smooth surface with sub-nanometer roughness is obtained. The obtained value of surface roughness is in good agreement with the calculated value determined using conventional grinding theory. Compared to a conventional polishing process with a colloidal SiO_2 slurry, the proposed method shows superior polishing efficiency without the need for a polishing pad. SEM observation of the core–shell particles shows that the particles have durability against the strong electric field between the electrodes.

1. Introduction

The demand for single-crystalline silicon carbide (SiC) wafers has been rapidly increasing with the recent development of high-performance SiC power devices that utilize their excellent electronic and thermal properties. SiC wafers are generally produced using the same process for fabricating conventional Si wafers: slicing ingots by wire sawing [1,2], grinding [3], mechanical polishing [3–5], and mirror finishing with a multiple stage. In the final finishing stage, a process called chemical mechanical polishing (CMP) employs loose abrasives in the form of a SiO_2 or CeO_2 slurry to obtain smooth SiC surfaces [6–8]. However, owing to the extremely high mechanical strength and chemical inertness of the material, the material removal rate of CMP for SiC is extremely low—two or three orders of magnitude lower than that of a conventional Si CMP process—which results in a much longer processing time. In a loose-abrasive process, the polishing pad, which is used as a tool to hold the abrasives in the slurry, plays a key role, along with the polishing slurry, to obtain a mirror-like surface. However, because the polishing pad is generally made of resin, which is much softer than the abrasives and materials to be polished, the wear caused by the friction between the polishing pad and the material or abrasives makes it necessary to periodically replace the pad [9–11]. This leads to an increase in the processing cost of CMP. In the CMP of SiC in particular, the wear of the polishing pad is a crucial issue because of the extremely high mechanical hardness of the material and the long polishing time required.

Extensive studies have been carried out to improve the material removal rate of SiC in the finishing process via plasma-assisted polishing [12,13] and electrochemical mechanical polishing (ECMP) [14,15]. However, plasma-assisted polishing requires a complex polishing system and high electric power supply to generate the plasma. As reported by Li et al. [14] and Deng et al. [15], ECMP of SiC wafers comprises the electrochemical oxidation of SiC followed by mechanical removal of the resulting oxide by abrasives. The material removal rate

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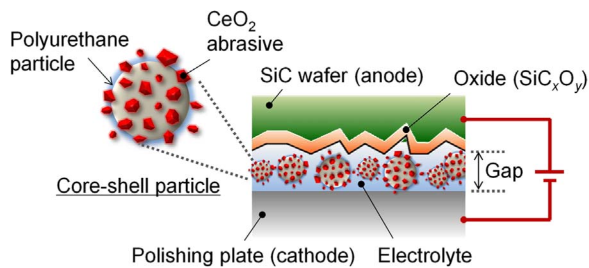

Fig. 1. Schematic view of the proposed ECMP method.

has been increased by introducing anodic oxidation to the inert SiC; however, previously reported ECMP processes required polishing pads to maintain a gap between the cathode and the SiC anode, and to hold abrasives in the slurry.

In this work, a novel technique that does not involve the use of a polishing pad is proposed for ECMP of SiC. As shown in Fig. 1, polyurethane–CeO_2 core–shell particles are employed as a polishing medium. The SiC surfaces are first oxidized by the electrochemical reaction between SiC and the polishing plate. The resulting oxide layer (SiC_xO_y) is then removed by CeO_2, which is much softer than SiC, on the core–shell particles. The core–shell particles play a key role in maintaining the gap between SiC and the cathode that is necessary for electrolysis. Although simple abrasives with a large diameter can also produce the gap, the obtained surface is rough. Furthermore, core–shell particles consisting of elastic core materials covered by soft and small abrasives prevent the generation of mechanical defects on the material surface because they inhibit direct contact between the polishing plate and the material. As a result, a smooth surface can be obtained without using a polishing pad. The use of polymer–abrasive core–shell particles in CMP processes has previously been proposed [16–20]. Chen et al. reported that when polystyrene–CeO_2 core–shell nanocomposites were used to polish a SiO_2 film in the CMP process, they showed a material removal rate and surface roughness that were superior to those obtained with conventional CeO_2 abrasives [20]. However, previously reported core–shell-structured abrasives were employed to polish soft material surfaces such as SiO_2 [16,19,20] and Cu [17,18], and there have been no reports on the CMP process with core–shell particles for hard and brittle materials such as sapphire, GaN, and SiC. Even when core–shell particles are employed, the CMP process is believed to be generally ineffective in polishing SiC surfaces because the abrasives (SiO_2 [16–18] or CeO_2 [19,20]) covering the core particles are much softer than the material.

In this paper, along with a report on the fabrication of polyurethane–CeO_2 core–shell particles, the polishing characteristics of the proposed ECMP method are described. The electrolysis conditions for obtaining a smooth surface were optimized by examining the electrolysis current, surface morphology, and chemical state of the polished surfaces.

2. Materials and methods

2.1. Preparation and characterization of core–shell particles

The core–shell particles were prepared by agitating polyurethane (PU) and CeO_2 particles at a controlled temperature in a dry atmosphere. The preparation process is described in detail elsewhere [21]. As shown in Fig. 2(a), the PU particles (Art Pearl U-600T, Negami Chemical Industrial, Japan) that were used as core particles had spherical shapes with an average diameter of approximately 10 μm. The shell material covering the PU particles was CeO_2 particles (SHOROX A-10, Showa Denko, Japan) with an average diameter of 1.4 μm (Fig. 2(b)). Several types of core–shell samples were prepared with different CeO_2/PU ratios (by weight). The mixed powders in the stainless vessel were agitated by a propeller stirrer and heated at a temperature of approximately 200 °C for 10 min. After cooling, the prepared samples were observed using a scanning electron microscope (SEM; SU1510, Hitachi High-Technologies, Japan) with a built-in energy-dispersive X-ray (EDX) spectrometer.

2.2. ECMP experimental apparatus and operating conditions

The polishing experiment was carried out using the prepared core–shell particles. The SiC samples used in this work were single-crystalline 4H-SiC $(000\bar{1})$ wafers (n-type; 50 mm (2 in) in diameter; on-axis) with resistivity of 0.01–0.03 Ω cm (TanKeBlue Semiconductor Co. Ltd., China). The surface of each sample was mechanically polished using 2.5-μm diamond abrasives with a cast iron polishing plate and a typical initial surface roughness (arithmetic average roughness, R_a) of 4–6 nm.

The polishing characteristics were evaluated using a single-sided polishing machine (FACT200, Nano Factor Co., Ltd., Japan), shown in Fig. 3, under the conditions summarized in Table 1. The polishing tool was a stainless steel plate with x–y grid grooves (width:2 mm; pitch:20 mm). Note that no polishing pad was attached to the polishing plate. An electric connection was formed between the SiC wafer and the polishing plate to enable anodic oxidation. As shown in Fig. 3(b), the SiC wafer was adhered to the insulating jig with a piece of double-faced tape (Intelimar SS4440NL2, Nitta Corp., Japan), on which a window was partly formed to allow electrical contact to the SiC wafer. The polishing slurry, which contained core–shell particles dispersed in deionized (DI) water, was magnetically stirred during polishing. The current flowing between the SiC wafer and the polishing plate was measured using a digital multimeter (DMM 2100, Keithley instruments, USA). After polishing followed by DI water rinsing, the sample surface was evaluated using an optical interferometric microscope (NewView 5032, Zygo, USA) over an area of 720×540 μm^2 and using EDX spectroscopy.

3. Results and discussion

3.1. Optimization of the process for fabricating core–shell particles

The weight ratio of CeO_2 to PU for producing the core–shell particles was optimized through SEM–EDX observations. As shown in Fig. 2(c), when the CeO_2/PU ratio was 2.0, bare PU surfaces were partly exposed even though the CeO_2 particles were integrated into the PU particles, indicating that there were insufficient CeO_2 particles to cover the PU particles. The absence of CeO_2 particles on the core PU surfaces led to the poor ability to remove the anodic oxide layer on SiC surfaces during the ECMP process. In contrast, when the CeO_2/PU ratio was set to 4.0 (Fig. 2(d)), the PU surfaces were entirely covered with CeO_2 particles. However, excess CeO_2 particles that were not integrated into the PU particles appeared around the core–shell particles. Such excess CeO_2 particles were much smaller than the core–shell particles, and thus they could not remove the surface material, resulting in the waste of high-cost rare-earth abrasives. Excess CeO_2 particles were observed even when the CeO_2/PU ratio was 3.0 (not shown). However, as shown in Fig. 2(e), when the CeO_2/PU ratio was 2.5, the number of residual CeO_2 particles was drastically decreased and the PU surfaces were still entirely covered by the CeO_2 particles. EDX elemental maps shown in Fig. 4 also demonstrate that elemental Ce was detected over the entire core–shell particle, while elemental C originating from the PU particles was located only on a small part of each particle. This indicates that the CeO_2 particles were uniformly dispersed over the PU particles. Based on these results, the optimum CeO_2/PU ratio for fabricating uniform core–shell particles was determined to be 2.5.

Similar structured particles containing a polymer core with CeO_2 shell can also be produced using the process reported by Chen et al. [19]. However, their process required complex and prolonged wet chemical treatment, which also includes dispersion under ultrasonic

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