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Novel Abrasive-Free Planarization of 4H-SiC (0001) Using Catalyst

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A new abrasive-free planarization method for silicon carbide (SiC) wafers was proposed using the catalytic nature of platinum (Pt). We named it catalyst-referred etching (CARE). The setup equipped with a polishing pad made of Pt is almost the same as the lapping setup. However, CARE chemically removes SiC with an etching agent activated by a catalyst in contrast to mechanical removal by the lapping process. Hydrofluoric acid which is well known as an etchant of silicon dioxide (SiO_2) that cannot etch SiC, was used as the source of the etching agent to SiC. The processed surfaces were observed by Nomarski differential interference contrast (NDIC) microscopy, atomic force microscopy (AFM), and optical interferometry. Those observations presented a marked reduction in surface roughness. Moreover, low-energy electron diffraction (LEED) images showed that a crystallographically well-ordered surface was realized.

Key words: Silicon carbide, polishing, etching, catalyst, hydrofluoric acid, platinum

INTRODUCTION

Silicon carbide (SiC) is an attractive material having a variety of applications because of its excellent electrical, thermal, and mechanical properties. In particular, it is expected to be used in high-performance electronic devices such as high-power, high-temperature, and high-frequency devices. However, the well-ordered SiC surface is difficult to prepare because of its mechanical hardness and chemical inertness. Some of the current preparation methods are mechanical polishing using diamond abrasive with a hard lapping plate,[1,2] chemical mechanical polishing (CMP),[3–5] and hydrogen etching at a high temperature.[6,7] The mechanical polishing method can flatten wafer surfaces but can also cause mechanical damage. Chemical mechanical polishing reduces surface damage, but cannot remove it completely. Moreover, waviness increases during the processing because elastically soft pads are employed in CMP. Hydrogen etching is an indispensable pretreatment method for epitaxial layer growth to remove crystallographic damage and contamination on substrates. However, the removal rate is not high and a large amount of electric energy is required to produce a high temperature.

In this paper, we propose a new abrasive-free planarization method using a catalytic reaction, which is called “catalyst-referred etching (CARE).” As mentioned above, the SiC planarization process has many sequences because SiC surfaces for electrical devices are required to have good flatness, low microroughness and low crystallographic damage. Wafer flattening methods such as lapping involve hard strong abrasion between a wavy wafer and a flat polishing plate through the preferential removal of the topmost wafer surface in contact with the flat polishing surface. The abrasive method deforms the surface crystalline structure owing to the mechanical removal that deteriorates microroughness. In contrast, hydrogen etching using chemical reaction removes SiC without surface damage but cannot efficiently improve flatness because the wafer surface is randomly etched without reference to a flat surface. Chemical mechanical polishing has both characteristics of chemical removal and mechanical removal. Wafer waviness increases owing to the use of elastically soft pads in CMP; however,

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microroughness is reduced because the removal mechanism is a mechanically assisted chemical reaction between the powder particles and the wafer surface.

We supposed that the most efficient planarization can chemically remove and simultaneously realize a flat surface, which can be achieved using a catalyst as the polishing plate. If the catalyst generates reactive species that are activated only next to the catalyst surface and deactivated when leaving the surface, the flat surface can be realized by chemical removal. We performed an initial experiment in which a platinum (Pt) wire as catalyst was relatively moved in contact with SiC in hydrogen fluoride solution. The result showed that hard SiC can be removed. The reason for using Pt and HF is as follows. The Pt has catalytic properties such as the dissociation of various molecules,[8] and the interaction between Pt surfaces and hydrogen fluoride molecules in aqueous atmosphere can generate F and OH by self-dissociation. These species chemically removed SiC and Si in aqueous solution[9,10] or in plasma.[11]

EXPERIMENTAL SETUP

CARE is applicable to SiC planarization from flattening a wafer to surface finishing for electric devices. The CARE planarization setup has a polishing pad and a sample holder that rotate similarly to those in lapping. The pad is made of solid Pt, flattened by cutting work, and not polished. Therefore, the roughness of the pad surface is not good but does not influence sample roughness finishing—the averaging effect by two-axis rotations that changes the contact point between the pad and the sample thus improving microroughness. The pad and the sample are immersed in hydrofluoric acid, which is the reactive species source in the process. Hydrofluoric acid can etch some materials, for example, SiO_2, but cannot etch SiC only by dipping.

EXPERIMENTAL CONDITIONS

The CARE setup has a catalytic polishing pad made of Pt that is placed on the rotational table. Wafers are attached on the sample stage using wax and are placed on the pad with a controlled pressure of 0.02 MPa, which is noticeably lower than that in the conventional polishing process. The polishing pad and the sample in the process are dipped in 2 mol/L hydrofluoric acid. The rotation speeds of the sample stage and the pad were fixed at 20 and 19 rpm, respectively. Removal rates of 0.1–0.2 μm/h can be obtained under such conditions. The employed SiC wafers (4 H n-type) were commercially available and have resistivities and micropipe densities of 0.02–0.03 Ωcm and 50–100 cm^{-2}, respectively. The wafers were cut and lapped to expose the (0001) Si plane on-axis. The processed surfaces were first observed by Nomarski differential interference contrast (NDIC) microscopy. Furthermore, the surface qualities were evaluated by optical interferometry (ZYGO Corp., Middlefield, CT NewView 200 CHR), atomic force microscopy (AFM, SII Nanotechnology, Inc., Chiba, Japan, SPA400 + SPI3800N), and low-energy electron diffraction (LEED) analysis.

RESULTS AND DISCUSSION

The NDIC images of CARE surfaces are shown in Fig. 1b and c. As-lapped surfaces were also observed (Fig. 1a). On the as-lapped surface, many scratches were observed in the 880 μm × 1,140 μm image (Fig. 1a), and many microcracks were observed in the 60 μm × 80 μm image. Such scratches accompanying microcracks were introduced during lapping. The lapping process generated and accumulated scratches and cracks on the surface so that a defect-free surface could not be obtained. In contrast, the CARE chemically removed surface roughness through the catalytically activated chemicals existing on the pad surface. Therefore, the topmost sites of the wafer surface in contact with the pad surface were preferentially removed. Mechanical scratches or cracks were not introduced as long as the frictional force between the surfaces was sufficiently small.

The CARE surface with a removal depth of 3 μm from the lapped surface and the surface of a commercially obtainable SiC wafer prepared by conventional CMP were observed by optical interferometry and are compared in Fig. 2a and b. There are no scratches on the CARE surface compared with the CMP surface. The maximum height of the surface irregularity (P-V) and root-mean-square roughness (RMS) were markedly lower than those on the CMP surface and evaluated to be 1.236 nm and 0.142 nm,

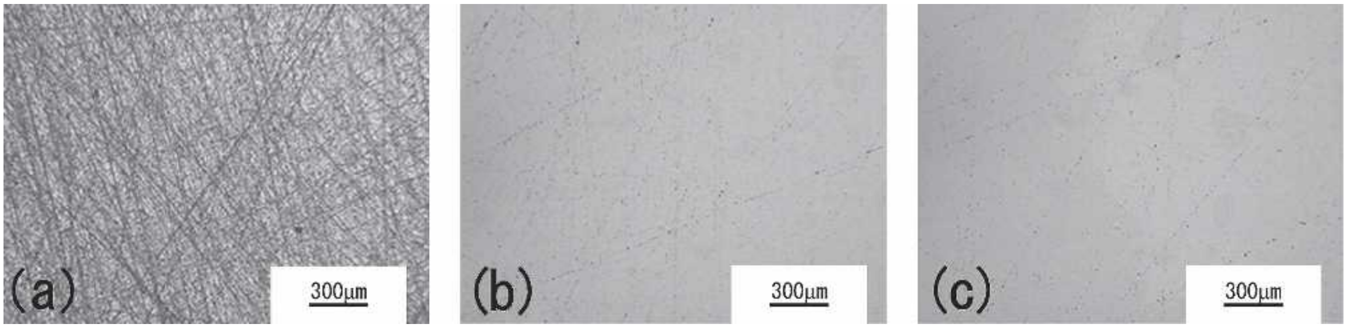

Fig. 1. NDIC images of 4H-SiC wafer: (a) as-lapped surface, (b) CARE-processed surface with removal depth of 1 μm, and (c) CARE-processed surface with removal depth of 2 μm. Many scratches were markedly reduced by CARE in the 880 μm × 1140 μm area.

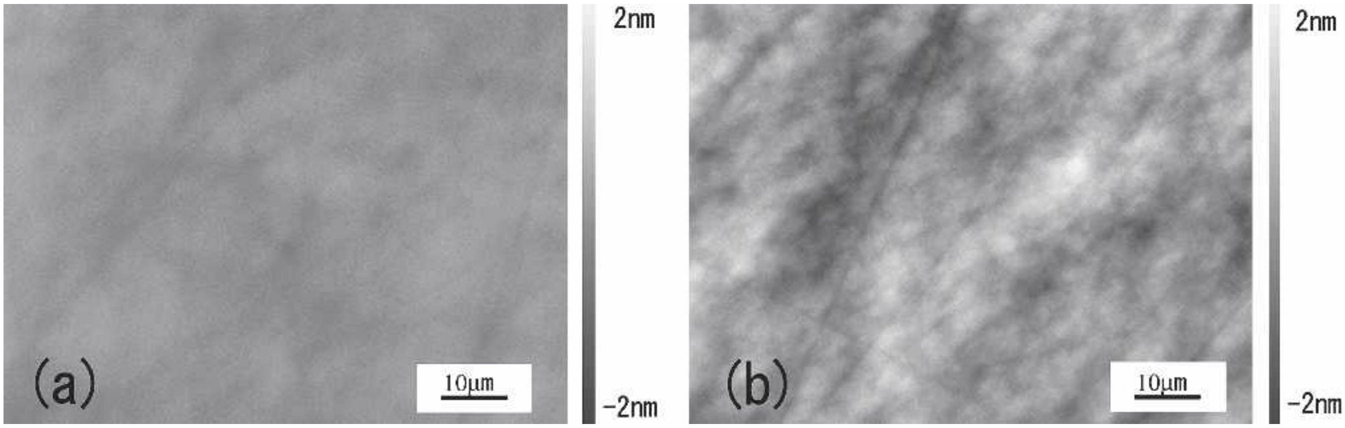

Fig. 2. Optical interferometer images of a 4H-SiC wafer: (a) CARE-processed surface with removal depth of 3 μm; RMS: 0.142 nm [P-V: 1.236 nm, Ra: 0.114 nm], and (b) as-received (after CMP) surface; RMS: 0.548 nm [P-V: 3.856 nm, Ra: 0.436 nm].

respectively. The CARE process can be expected to have a higher potential as a planarization method than conventional CMP.

To estimate the crystallographic properties of the CARE surface, CARE and CMP surfaces were observed by LEED with a probe beam energy of 65 eV corresponding to an estimation thickness of 0.4 nm (Fig. 3). LEED is a method used for estimating crystal structures on a surface. When the electron beam penetrates a well-ordered crystalline structure on the surface, a sharp diffraction spot is observed. The estimated thickness is determined from electron penetration depth. Sharp LEED spots are seen in the CARE surface (Fig. 3a) compared with the CMP surface in which only diaphanous and blurry spots are observed under such low-energy probe beam conditions (Fig. 3b).

The surface morphology of the processed surface was measured by AFM (Fig. 4). The measured area was 1 μm ×1 μm. A step-terrace structure was observed with a step height of approximately 3Å corresponding to one bilayer thickness of Si and C. Such a surface structure strongly suggests that the removal mechanism is based on chemical phenomena and that a type of step-flow removal occurs on the SiC (0001) Si surface.

The above results show that CARE can produce a smooth surface of 0.1 nm (Ra 0.114 nm RMS 0.142 nm) level roughness. From the AFM and LEED observations, it can be seen that the crystalline structure of the CARE surface is well ordered and damage free. However, the process mechanism has not yet been clarified. Although SiC surfaces cannot be etched by hydrofluoric acid, a SiC surface in contact with a Pt surface is chemically etched in this process. At the interface between the SiC and Pt surfaces, various mechanochemical and catalytic phenomena are induced and contribute to the removal of surface

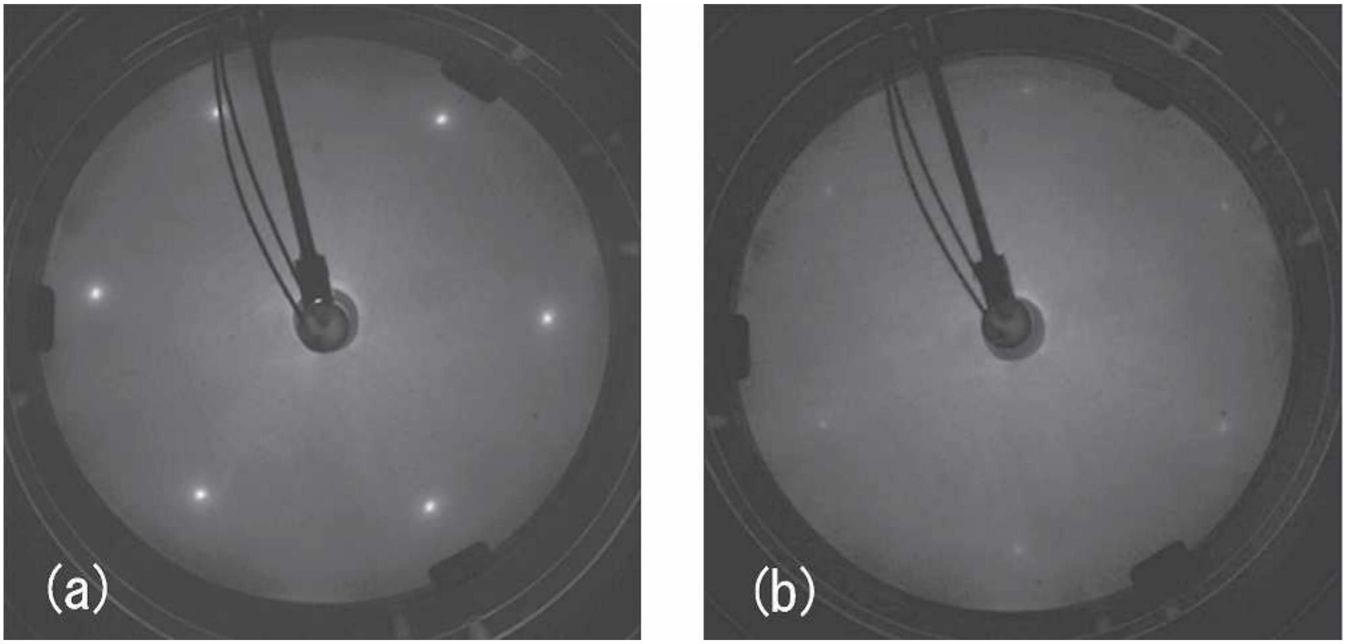

Fig. 3. Photographs of LEED spot with probe beam energy of 65 eV corresponding to estimated thickness of 0.4 nm: (a) CARE-processed surface with removal depth of 3 μm and (b) as-received (after CMP) surface. Sharp LEED spots, which show a crystallographically well-ordered surface, are observed in the CARE surface.

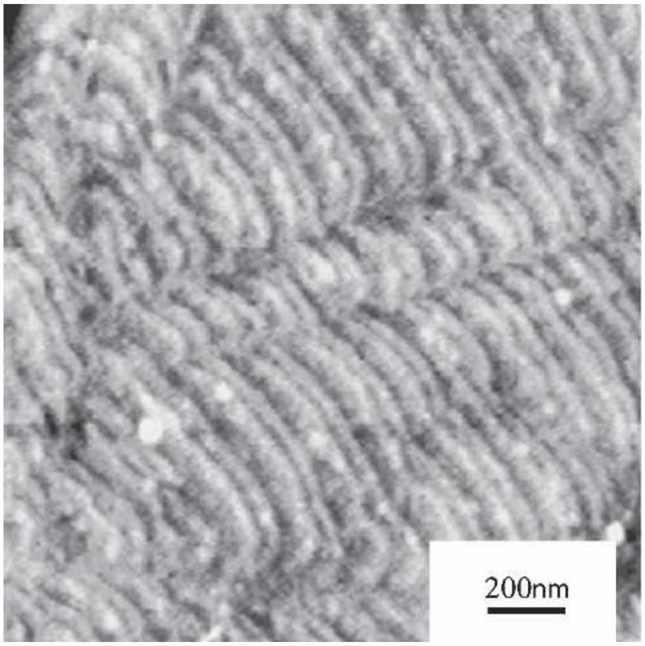

Fig. 4. AFM images of CARE-processed surface with removal depth of 3 μm in 1 μm × 1 μm area; RMS: 0.118 nm [P-V: 2.649 nm, Ra: 0.089 nm].

atoms on the SiC crystal surface. We investigated the ability of etching for a SiC surface using a polycarbonate pad instead of Pt with the same contact pressure as that of a Pt pad and observed no removal of SiC surfaces. This fact clearly shows that the removal mechanism is not based only on mechanical phenomena but that the catalytic nature of Pt also plays a critical role. The details of the reaction at the interface should be clarified in future research.

CONCLUSIONS

We proposed a new abrasive-free planarization method for SiC wafers that was named CARE. CARE planarization uses a Pt polishing pad as catalyst, which activates hydrofluoric acid to reactive species and removes SiC chemically. Optical microscopy and atomic force microscopy (AFM) images showed a marked reduction in surface roughness compared with CMP. Furthermore, low-energy electron diffraction observation showed that a crystallographically well-ordered surface was realized. Our results show that CARE is a more efficient planarization process than CMP.

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