High-integrity Finishing of Reaction Sintered SiC by Plasma Assisted Polishing Using Ceria Abrasive

K. Yamamura, M. Ueda, H. Deng, N. Zettsu
Research Center for Ultra-precision Science and Technology, Graduate School of Engineering, Osaka University, Japan
yamamura@upst.eng.osaka-u.ac.jp

Abstract

Reaction-sintered (RS) SiC is a useful material for a high-performance molding die of a glass lens or a highly rigid lightweight structure and so on. However, the high-integrity finishing of the SiC material is difficult because of the high hardness and chemical inertness of such a material. We proposed a new finishing technique, named plasma assisted polishing (PAP), which combines oxidation by atmospheric pressure water vapor plasma and abrasive polishing. PAP using a CeO_2 abrasive decreased both the number and depth of scratches on the surface compared with the surface polished by a diamond abrasive. Under a low RF power supply condition (12 W), protrusions with a height of 10-20 nm were formed on the surface. In contrast, an increase in RF power (30 W) resulted in the absence of residual protrusions and rms roughness was improved from 4.63 to 2.31 nm.

1 Introduction

Silicon carbide (SiC) is a promising next-generation semiconductor material because of its excellent electrical, chemical, thermal and mechanical properties. With different manufacturing technologies, different types of SiC are obtained, such as single-crystal SiC grown by a modified Lely method, polycrystalline SiC prepared by chemical vapor deposition (CVD) and sintered SiC. In particular, reaction-sintered (RS) SiC shows many excellent properties, such as high bending strength and high thermal conductivity, compared with conventionally sintered SiC.[1)] These properties make RS-SiC one of the most attractive materials for a glass lens mold, equipment parts for the fabrication of semiconductor devices, and a lightweight space telescope mirror substrate. However, it is difficult to obtain a high integrity surface for RS-SiC because of its high hardness and chemical inertness. There are several approaches to preparing a SiC wafer with a smooth surface. Chemical mechanical polishing (CMP) is now widely used as a finishing process for the surfaces of single-crystal SiC and

GaN substrates.[2)] Catalyst-referred etching (CARE), which was proposed by Yamauchi *et al.*, enables us to obtain an atomically flat surface of 4H-SiC.[3)] Other techniques such as the photochemical polishing technique proposed by Watanabe *et al.*, are under development. [4)] However, all of these techniques are focused on the finishing of single-crystal SiC, and the material removal rate of CMP, which is reported to be less than 0.5 μm/h, is very low. Although a high rate of mechanical polishing with a hard abrasive, such as diamond, is achieved, microscratches and subsurface damage are inevitably introduced. To resolve the above issues, we proposed a new finishing technique, named *plasma assisted polishing* (PAP), which combines oxidation by atmospheric pressure water vapor plasma and abrasive polishing.[5)] It is expected that oxidized soft layer polishing using a soft abrasive will lead to scratch and damage-free surfaces of difficult-to-machine materials. The aim of this study is to evaluate the chemical and morphological structures of the RS-SiC surface processed by PAP.

2 Experimental setup and parameters

Figure 1 shows a schematic of the experimental apparatus used for preliminary research. This apparatus consists of separately installed plasma generation and mechanical removal parts, which are used to investigate the basic removal mechanism. Atmospheric pressure plasma is generated by applying an RF (f =13.56 MHz) electric power, and helium-based water vapor (1.7-2.6%) with a flow rate of 1.5 L/min is supplied as a process gas by bubbling helium through ultrapure water (UPW), and its concentration is measured using a dew-point meter (DPM). The copper electrode used is covered with a closed-end quartz glass tube to prevent arc discharge through the generation of dielectric barrier discharge (DBD). In the mechanical removal part, polishing using a polishing film (φ8 mm) was conducted. The specimen was placed on a rotary table, and surface modification by plasma irradiation and mechanical removal using a polishing film were sequentially repeated. We used a commercially available RS-SiC substrate with a size of 30 mm x 30 mm x 2 mm^t as a specimen. Table 1 show the experimental parameters used.

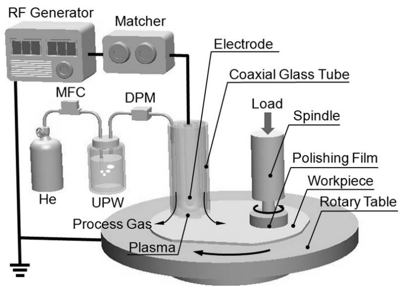


Figure 1: Schematic of experimental setup

Table 1: Experimental parameters

Load	54 g
Abrasive	Diamond, CeO_2, φ 0.5 μm
Rotation Speed	200 rpm (Pad), 120 rpm (Subst.)
Process Gas	He+H_2O (1.97%), 1.5 SLM
RF Power	Diamond: 0 W, CeO_2: 12 W, 30 W
Processing Time	60 min

3 Results and discussion

3.1 Surface chemical composition

Figure 2 shows the X-ray photoelectron spectroscopy (XPS) spectra of the RS-SiC surface before and after 10 min plasma irradiation; it also shows the (a) Si2p, (b) C1s and (c) O1s spectra, respectively. Before plasma irradiation (i), the strongest peaks (100.15 eV, 282.7 eV) that correspond to a Si-C bond indicate that the main component of the as-received surface is SiC. A Si-O bond (102.9-103.6 eV, 533.0 eV) is also observed; it may due to the high-temperature oxidation during the reaction sintering process. After plasma irradiation (ii), the SiC component disappeared, while the intensity of Si-O markedly increased. These results show that the RS-SiC surface was oxidized by water vapor plasma irradiation. In our previous study, a nanoindentation test revealed that the hardness of the oxidized RS-SiC surface (1.4 GPa) decreased by about one order of magnitude compared with that of the as-received one (15.6 GPa). [6)]

3.2 Surface morphology

Figure 3 shows the surface polished by a diamond abrasive without plasma irradiation. The p-v and rms roughness measured by microscopic interferometer are 26.28 nm and 3.33 nm, respectively. The SEM image shows that many scratches are formed on the surface. Figure 4(a) shows the surface polished by PAP using a ceria abrasive with a low RF power of 12 W. The roughness, whose p-v is 38.60 nm and rms is 4.63 nm, increased compared with that in the case of diamond abrasive polishing because many protrusions with a height of 10 nm were formed on the surface. This may due to the insufficient and nonuniform oxidation of RS-SiC, which includes various types of grains, such as α-SiC, β-SiC, residual silicon and carbon. [7)] However, the number of scratches decreased, as shown in the SEM image. Figure

4(b) shows the surface polished by PAP with a high RF power of 30 W. By increasing the RF power from 12 W to 30 W, protrusions on the surface disappeared owing to the sufficient oxidation, and the surface roughness decreased to 17.88 nm p-v

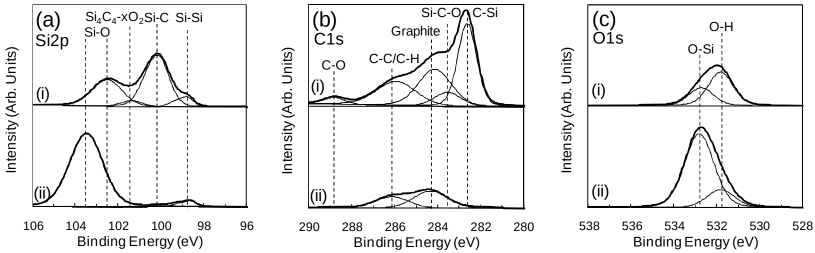


Figure 2: XPS spectra of (i) as-received and (ii) plasma irradiated RS-SiC surface

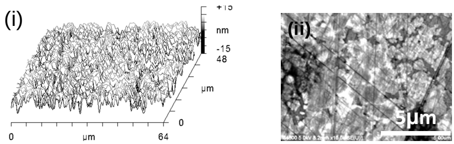
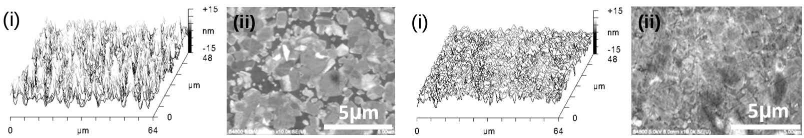
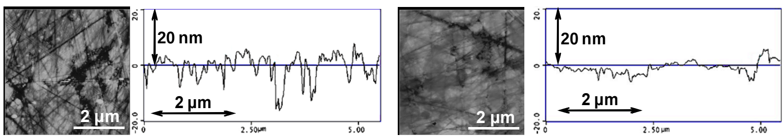


Figure 3: Surface morphologies of RS-SiC processed by diamond abrasive



(a) Low RF power (12W) (b) High RF power (30W)

Figure 4: Surface morphologies of RS-SiC processed by PAP using ceria abrasive



(a) Processed by diamond abrasive (b) Processed by PAP using ceria abrasive

Figure 5: AFM images of RS-SiC surface

and 2.31 nm rms. Figures 5(a) and (b) show the atomic force microscopy (AFM) images of the polished surface. In the case of diamond abrasive polishing (a), many scratches were observed on the surface, and their depths ranged from 10 to 20 nm. In contrast, in the case of PAP using a ceria abrasive, the number of scratches decreased and the scratches observed were very shallow. From these results, it is concluded that

PAP enables us to obtain a high-integrity surface for difficult-to-machine materials, such as RS-SiC.

Acknowledgments

This work was partially supported by a Grant-in-Aid for Scientific Research (B) (20360068) from the MEXT, Japan, a research grant from the Die and Mold Technology Promotion Foundation and a Mazak Foundation and a research grant from the Osawa scientific studies grants foundation.

References:

[1] S. Suyama, *et al.*, Diamond and Related Materials **12** (2003) 1201.

[2] L. Zhou, *et al.*, J. Electrochem. Soc. **144** (1997) L161.

[3] H. Hara, *et al.*, J. Electro. Mater. **35** (2006) L11.

[4] J. Watanabe, *et al.*, Proc. of ASAAT 2007 (2007) 91.

[5] K. Yamamura, *et al.*, Adv. Mater. Res. **126-128** (2010) 423.

[6] K. Yamamura, *et al.*, Proc. of 25th Annual Meeting of the ASPE (2010) 61.

[7] S. Suyama, *et al.*, Proc. of SPIE **6666** (2007) 66660K-1.