# Chemical state modification of 4H-SiC by ultraviolet-ray aided machining

Akihiro Hata<sup>1</sup>, Masaru Takizawa<sup>1</sup>, and Takeshi Tanaka<sup>2</sup>

1) Department of Physical Sciences, Faculty of Science and Engineering, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan

2) Research Organization of Science and Technology, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan

# Abstract

An ultraviolet-ray aided machining (U-RAM), which attempts to add a chemical reaction through ultraviolet-ray irradiation to a mechanical polishing, is a promising procedure to obtain a flat surface of SiC in a short time. In order to investigate how chemical states change in the U-RAM process, we have performed X-ray absorption fine structure measurements. As a result, it was found that a mixed compound other than SiO<sub>2</sub> was produced on the SiC surface by the U-RAM. We think that polishing efficiency of U-RAM is better than ordinary mechanical polishing because of generating the mixed compound of Si, C and O on the surface.

#### 1. Introduction

A SiC has attracted much attention as a substrate for electronic devices. In order to efficiently obtain a flat surface of SiC, many polishing procedures have been performed. Among them, an ultraviolet-ray aided machining (U-RAM) [1] is a promising procedure to obtain a flat surface of SiC in a short time, where an irradiated photocatalyst and fluorescent substance would provide an electron and a hole to act as oxidation/reduction reactions to the surface and a photocatalyst would also scratch the surface mechanically with abrasive grit. Previous work on the U-RAM of Al has revealed the oxidation of the Al surface [2]. Although applying the U-RAM to 4H-SiC improves a polishing rate, it is not clear how the surface of SiC is oxidized. In order to study the chemical state changes, we have performed X-ray absorption fine structure (XAFS) measurements of SiC during each U-RAM procedure.

## 2. Experimental

#### Sample preparation

We prepared a 4H-SiC with a flat surface (commercial SiC) and then sandblasted commercial SiC to obtain a SiC with a rough surface (sandblast SiC). An U-RAM of this sandblast SiC was performed using a TiO<sub>2</sub> particle (~7 nm) as a photocatalyst and a Cathilon dye as a fluorescent substance under the irradiation of UV lamp ( $\lambda = 253.7$  nm) with diamond grit (0-20 µm), referred as U-RAM SiC. Furthermore, we continued two kinds of U-RAM procedures by exchanging grits; one with CeO<sub>2</sub> grit (U-RAM SiC further polished with CeO<sub>2</sub>), the other with smaller-size diamond grit (5 nm) [U-RAM SiC further polished with Dia (5 nm)]. In order to see an effect of the slurry and the UV irradiation, we only immersed sandblast SiC and commercial SiC in the TiO<sub>2</sub> and the Cathilon dye (immersed sandblast SiC, immersed SiC) under UV irradiation (immersed sandblast SiC + UV). As a reference, SiO<sub>2</sub>/Si sample was also prepared.

## Depth-resolved XAFS measurements

XAFS measurements were performed at the BL-8 of SR Center at Ritsumeikan University, equipped with a grazing incidence monochromator with a varied-line-spacing plane grating. In order to obtain the depth-resolved information, XAFS spectra of the samples were simultaneously collected by total electron yield (TEY) and total fluorescence yield (TFY) through a sample drain current and a micro-channel plate detector with retarding grids, respectively. TEY spectra have the information near the surface (1-5 nm), while TFY spectra have the information mainly inside the bulk (more than 10 nm). The retarding voltage was set to -150 V for Si *L*-edge. In addition, setting the retarding voltage to -50 V enabled us to

obtain more surface-sensitive (less than 1 nm) XAFS spectra by partial electron yield (PEY). The measurements were performed at room temperature under ultrahigh vacuum of  $\sim 1 \times 10^{-7}$  Pa. The incident angle of SR with respect to the surface normal was set to 0°.

## 3. Results and Discussion

Figure 1 shows the Si *L*-edge TEY spectra of commercial SiC, U-RAM SiC, sandblast SiC, and SiO<sub>2</sub>/Si. For commercial SiC, the onset appears around 101.8eV, which is a characteristic feature of 4H-SiC [3]. The TEY spectrum of U-RAM SiC is different from that of commercial SiC, that is, a prominent feature starts from ~103.5 eV though the onset also appears around 101.8eV. For sandblast SiC, the onset also appears around 101.8eV and a prominent feature appears around 108 eV. As seen in the spectrum of SiO<sub>2</sub>/Si, the structures around 106 eV and 108 eV come from SiO<sub>2</sub> and the structure around 101 eV comes from Si. From these results, the surface of sandblast SiC is mainly covered with SiO<sub>2</sub> due to the high energy collisions, and on the surface of U-RAM SiC exists not SiO<sub>2</sub> but other compounds besides SiC.



**Fig. 1** Si *L*-edge TEY XAFS spectra of 4H-SiC during each U-RAM procedure. A reference SiO<sub>2</sub>/Si XAFS spectrum is also shown.

Figure 2 shows the Si *L*-edge TFY spectra of commercial SiC, U-RAM SiC, and sandblast SiC. The TFY spectra of U-RAM SiC is similar to that of commercial SiC, indicating that the chemical state of U-RAM SiC in the bulk is SiC. For sandblast SiC, the structure of SiC starting from ~101.8 eV is clearly seen, also indicating that the chemical state of sandblast SiC in the bulk is SiC, though the structures of SiO<sub>2</sub> (around 106 eV and 108 eV) still appears maybe due to the rough surface.



Fig. 2 Si L-edge TFY XAFS spectra of 4H-SiC during each U-RAM procedure.

Since other compounds were found on the surface of U-RAM SiC, we further polished U-RAM SiC by exchanging grits. Figure 3 shows the Si *L*-edge TEY spectra of commercial SiC, U-RAM SiC further polished with Dia (5 nm), U-RAM SiC further polished with CeO<sub>2</sub>, and U-RAM SiC. The TEY spectra of U-RAM SiC further polished with Dia (5 nm) and U-RAM SiC further polished with CeO<sub>2</sub> is similar to that of commercial SiC. The surfaces are confirmed to be 4H-SiC free from the newly formed compound starting from ~103.5 eV. Because SiC is harder than CeO<sub>2</sub>, CeO<sub>2</sub> grit could polish not SiC but the newly formed compound, suggested to be a brittle compound compared to SiC.



Fig. 3 Si L-edge TEY XAFS spectra of 4H-SiC for other U-RAM procedures.

Figure 4 shows Si *L*-edge TEY spectra of sandblast SiC, immersed sandblast SiC, and immersed sandblast SiC + UV. For the TEY spectra of immersed sandblast SiC and immersed sandblast SiC + UV, the structure around 103 - 106 eV increases, indicating that the newly formed compound appears on the surface of sandblast SiC after the immersion and/or UV irradiation.



Fig. 4 Si L-edge TEY XAFS spectra of sandblast SiC with the immersion and UV irradiation.

Figure 5 shows Si *L*-edge PEY spectra of commercial SiC, immersed commercial SiC, and immersed commercial SiC + UV. The spectra hardly change. This suggests that only rough SiC surfaces are affected by the immersion and/or UV irradiation. Then, further polishing with CeO<sub>2</sub> and smaller-size diamond grits does not produce the newly formed compound because of the flat surface.



Fig. 5 Si L-edge PEY XAFS spectra of commercial SiC with the immersion and UV irradiation.

Next, we consider the newly formed compound. We think that the compound is a mixture of Si, C and O. Because, it is thought that oxidation occurs on the surface of SiC, and from the previous research [4] it is confirmed that mixed compounds of Si, C and O are produced by a thermal treatment. Although the newly formed compound is not clear at present, we think that polishing efficiency of U-RAM is better than ordinary mechanical polishing because of produced mixed compounds of Si, C and O on the surface.

## 4. Conclusions

We have analyzed chemical state of 4H-SiC samples by XAFS. Not SiO<sub>2</sub> but mixed compounds exist after U-RAM. We think that polishing efficiency of U-RAM is better than ordinary mechanical polishing because of generating the mixed compound of Si, C and O on

the surface.

# References

[1] T. Tanaka, J. JSAT. 59, 402 (2015) (in Japanese).

[2] Takeshi TANAKA, MEMOIRS OF THE SR CENTER RITSUMEIKAN UNIVERSITY No. 17, 95 (2015).

[3] Y.F. Hu, H. Piao, J. Fronheiser, K. Matocha, J. Electron Spectrosc. Relat. Phenom. 184, 245–248 (2011).

[4] B. Hornetz, H-J. Michel and J. Halbritter J. Mater. Res., 9, 3088 (1994).