Investigation into electrochemical oxidation behavior of 4H-SiC with varying anodizing conditions

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ARTICLE INFO

Keywords:
- 4H-SiC
- Electrochemical oxidation
- Anodizing conditions
- Anisotropy
- Electrolytes
- Electrochemical-assisted hybrid machining

ABSTRACT

Electrochemical-assisted hybrid machining has been widely utilized for processing of single crystal SiC, which is a very promising next-generation semiconductor material for high power, high frequency and high temperature applications. With the aim of optimizing the machining process to achieve both high efficiency and high surface integrity, the electrochemical oxidation behaviors of 4H-SiC were experimentally investigated with varying the anodizing conditions from the aspects of electrolyte types, ionic concentration, potentials etc. in this study. Experimental results demonstrated that neutral solutions showed higher oxidation ability of 4H-SiC in comparison to acidic and alkaline electrolytes. 4H-SiC could be electrochemically oxidized in an extremely low concentration electrolyte, and mass concentration 1–2% could achieve highest film thickness of 197 nm and initial oxidation rate of 4.9 nm/s. The oxidation rate increased exponentially at the initial stage of anodization before reaching saturation. Meanwhile, the oxidation process exhibited significant anisotropy, with the oxidation rate of C-face 5–15 times higher that of Si-face. On the other hand, the hardness of the electrochemically-oxidized 4H-SiC surface was reduced by more than 90%. It was also noticed that the micro morphology of oxidized surface was significantly influenced by the ion types in the electrolytes. Furthermore, the electrochemical oxidation mechanism of 4H-SiC were discussed based on the experimental findings. The OH⁻ radical was considered the main factor determining the oxidation process of SiC. The research results deepen the understanding of the electrochemical oxidation of SiC and provide insights for optimizing the electrochemical-assisted hybrid machining process.

1. Introduction

Single crystal silicon carbide (SiC) possesses superior electrical and physical properties over Si, such as a wider band-gap, higher thermal conductivity, higher breakdown electric field, and higher saturation electron drift speed, etc. [1,2], which make it the ideal material for the energy-efficient third-generation semiconductors, especially for power devices that operate under high-temperature, high-voltage and high frequency conditions [3–5]. As a substrate material for electronic components, fabrication of SiC wafer requires both high shape accuracy and high surface integrity with no subsurface damage (SSD) [6]. On the other hand, single crystal SiC is a well-known difficult-to-machine material due to its extremely high hardness, brittleness and chemical inertness, which results in a high machining cost and low throughput. This has significantly hindered the development and widespread application of the SiC semiconductors. In order to improve both the efficiency and the resulted surface quality [7], electrochemical mechanical hybrid machining methods have been widely proposed and applied in fabrication of SiC and SiC-like materials, such as electrochemical-assisted multi-wire saw [8], electrochemical mechanical grinding, electrochemical mechanical polishing (ECMP), etc. [6,9]. In these methods, the workpiece surface is modified by electrochemical oxidation to decrease the material hardness and brittleness before or during the machining process to enable easier material removal [9–11]. In addition, low or zero SSD can be achieved owing to the existence of oxidation layer in the process. Except for the cases of ultra-precision machining process which require an atomic level precise control of the oxidation process, a higher oxidation rate is generally expected to realize more efficient machining. Nevertheless, few researches have been done on investigation and optimization of the electrochemical oxidation process.

In previous researches, the electrochemical oxidation mechanism of SiC has been emphatically studied. X. Yang et al. [12] demonstrated that the growth of oxide protrusions was controlled by the charge transfer process at the initial stage, which changed to a diffusion process after the oxide protrusions grew to a size of 100 nm order. N. Liu
et al. [13] pointed out that the oxidation process was non-uniform and local oxidation occurred on the charge carrier-rich areas such as damaged areas and doping sites. M. Herrmann et al. [14] found that anodic oxidation and active dissolution occurred on SiC ceramics in alkaline solutions and the SiC would convert to SiO$_3^{2-}$. D. H. van Dorp et al. [15] studied electrochemical etching of 4H-SiC in HF solution and concluded that the oxide formed at positive potential was much thicker for SiC than for Si. X. Yang et al. [16] used ultrasonic vibration on the anodic oxidation of 4H-SiC to enhance the oxidation rate by about 34% in the initial oxidation stage. On the other hand, there has been little investigation of the oxidation behavior of SiC under different electrochemical conditions and its optimization. In this study, with the aim to improve the machining performance of electrochemical-assisted hybrid machining process, the anodic oxidation of 4H-SiC with varying electrochemical conditions in aspects of electrolyte ions, ion concentrations and anodic potentials etc. are investigated. Furthermore, based on the experiment results, the electrochemical oxidation mechanism of SiC was discussed and approaches for optimizing the electrochemical-assisted hybrid machining was proposed. Within this paper it was focused on 4H-SiC only, but the techniques presented can also be applied to other material similar to SiC.

2. Material and experimental methods

On-axis, n-type 4H-SiC wafers (TankeBlue Semiconductor Co. Ltd.), with a thickness of 350 μm and a specific resistance of 0.015–0.028 Ω·cm were used as the specimen in this work, of which the C-face is mechanically polished, and Si-face is polished by chemical mechanical polishing (CMP). Before each experiment, the SiC specimen was degreased by sonicating in acetone for 4 min, followed by rinsing with deionized water and drying in the air.

A three-electrode electrochemical system, as shown in Fig. 1, was employed to study the anodizing properties of SiC. The 4H-SiC wafer was set as the working electrode (WE), while the counter electrode (CE) and the reference electrode (RE) was a platinum electrode and calomel electrode, respectively. In the experiment, SiC wafer was held by a specially-made electrode holder to make sure that only the specimen part was in contact with the electrolyte to avoid stray current. The anodizing experiments were carried out using a potentiostat (CorrTest CS2350H). Five different types of electrolyte solutions were used in the experiments for comparative study. The specific experimental conditions are shown in Table 1.

The surface morphology of 4H-SiC after anodizing was observed using atomic force microscopy (AFM, Bruker Edge). The generated oxide film thickness and hardness was measured with an ellipsometer (Film Sence-1) and a nanoindentation tester (Hysitron Ti-950), respectively. The change of surface oxygen content after oxidation was characterized using energy dispersive X-ray spectroscopy (EDX, Oxford x-stream-2). The chemical composition was analyzed using X-ray photoelectron spectroscopy (XPS) (ULVAC-PHI 5000 Versaprobe III) with an Al Kα radiation source.

3. Results and discussion

3.1. Anodic polarization curve

The anodic polarization curves of 4H-SiC in different electrolyte solutions were examined by applying a dynamic scanning potential to determine the passivation behavior. The results are shown in Fig. 2. For comparative analysis, the polarization curves of carbon steel (Q235) were also presented, which differed significantly from 4H-SiC. By contrast, SiC showed no clear passivation region, and the overall current density was three orders of magnitude less than carbon steel due to its high resistivity. At the early stage of anodization within a low voltage range, SiC was in an activation state and the current density kept to increase constantly. Further increasing the potential resulted in a passivation region and the current density curve fell. However, the polarization curve went up again after that, followed by fluctuations with multiple peaks, indicating a transition state between passivation and transpassive dissolution. On the other hand, the equilibrium potential in alkaline solution is always lower than the neutral and acidic solutions due to higher concentration of OH$^-$. In addition, the current density in NaOH solution was the highest, followed by neutral solution and the acidic solution was the lowest. The reason for this phenomenon was considered that the conductivity of NaOH solution was higher and 4H-SiC was more activated in alkaline electrolyte probably due to a more significant transpassive dissolution. At the end, the current densities of the five solutions became close due to the formation of the oxide film.

3.2. Anodizing behavior and surface properties

3.2.1. Oxidation film formation

By applying an anodic potential pulse to the 4H-SiC specimen, an oxidation film can be formed on the surface. The growth of oxidation film and the recorded trace of current transient for a potentiostatic potential pulse of 3.5 V in NaNO$_3$ solutions is shown in Fig. 3. It can be seen that the oxide film thickness increased exponentially at the beginning stage of oxidation, indicating an intense passivation process. The growth rate decreased after that due to the increase of the resistance of the oxidation film. This could be verified by the sharp decline of the corresponding current density. In comparison, the growth rate of oxide film was much lower in the lower concentration electrolyte, which was in accordance with the current density change. Based on the above results, it is concluded that the oxidation film rapidly grows at the initial stage of anodization with the time order of second and became saturated thereafter.
3.2.2. Anisotropy of oxidation

As a single crystal, 4H-SiC shows anisotropy in physical and chemical properties, which exerted great influence on the oxidation process. In the experiments, it was found that the oxidation degree between the C-face (0 0 0 –1) and the Si-face (0 0 0 1) showed a significant difference. As shown in Fig. 4, C-face presented an oxidation rate 5–15 times higher than that of Si-face under the same process conditions, especially when the anodization potential was higher. The mechanism of the phenomena is discussed in Section 4. After oxidation, the surface hardness decreased dramatically by more than 90% owing to the formation of oxidation layer, as shown in Fig. 5. In addition, the hardness of Si- and C-face after oxidation became almost equal, despite that the initial hardness of unoxidized Si-face was 10 GPa higher than C-face. This provides a notable advantage for high efficiency hybrid machining of 4H-SiC with no need to consider its crystalline anisotropy.

![Fig. 2. Comparison of polarization curves in different solutions. (a) 4H-SiC; (b) carbon steel.](image)

![Fig. 3. Growth of oxidation film with time in NaNO₃ and corresponding change of current density.](image)

![Fig. 4. Variation of surface oxidation film at different potentiostatic potentials. (a) Oxidation film thickness; (b) oxygen content.](image)

![Fig. 5. Change of surface hardness before and after anodizing.](image)
3.2.3. Surface morphology and the effect of applied voltage

Based on the above results of polarization curve and oxidation process, experiments on potentiostatic polarization of 4H-SiC at different voltages were conducted to investigate the anodization behavior by examining the surface micro-morphology and the oxidation degree.

Fig. 6 shows the optical photograph of anodized C-face in different electrolyte solutions. The surface was colored due to the formation of transparent oxide film. In addition, the color was constantly changing from 2.5 V to 9 V, indicating that the oxide film thickness gradually increased with voltage. Moreover, the initial voltage required for realization of coloring in different electrolyte solutions was different. Specifically, coloring phenomenon of 4H-SiC occurred at 2.5 V–3.5 V in neutral solution, 5 V in alkaline solution and 7 V in acidic solution, indicating 4-SiC was most easily oxidized in neutral electrolyte solution. With 7 V and 9 V, the oxide film was partly damaged with the occurrence of pit corrosion, as shown in Fig. 7, which was considered to be resulted from the transpassive dissolution under excessive current density.

The AFM-imaged micro morphology of the specimen surface after anodizing in neutral, alkaline and acidic solutions are shown in Figs. 8–10, respectively. It can be seen that the surface micro morphology varied considerably with the change of applied voltage, regardless of the electrolyte type. As the voltage increased, the appearance of the oxide film changed from loose to dense, indicating a more intensive oxidation reaction because higher voltage enhanced the charge transfer amount per unit time. In neutral solution, oxidation occurred preferentially at the scratches on the wafer surface at low voltages. When the voltage was sufficiently high, for example above 5 V, the whole surface was fully covered by a dense oxide film. In alkaline and acid solutions, however, the SiC was in active state at low voltages and oxide layer could hardly be observed on the surface, which was in accordance with the polarization curve. When the voltage exceeded a certain value, a compact film was formed on the whole surface evenly, as shown in Fig. 9(d–f) and Fig. 10(f). On the other hand, the electrolyte types also showed significant impact on the surface morphology of the oxide film, implying that electrolyte ions determined the oxidation to some extent. Specifically, round-shape oxide protrusions were generally observed in a neutral solution, as shown in Fig. 8(e). Instead, strip-shaped protrusions appeared in acidic solution (Fig. 9(f)), while the protrusions in alkaline solutions were cluster-like shape (Fig. 10(f)).

To further investigate the difference, the elemental chemical composition of the surface after oxidation was analyzed using XPS technique. Fig. 11 show the XPS spectrum analysis result of 4H-SiC surface after oxidation in different electrolytes. The colored lines represent the actually measured energy peaks, and the black curves represent the peaks of various chemical bonds after peak separation based on Gaussian-Lorentz function. In NaNO₃, no presence of Si-C bond could be confirmed after oxidation, indicating a complete oxidation that the whole surface was covered by oxides. On the other hand, Si-C bond
Fig. 8. Surface morphology change of C-face during anodizing in NaNO$_3$ solutions observed by AFM.

(a) 0V RMS:0.601nm  (b) 2.5V RMS:1.22nm  (c) 3.5V RMS:8.80nm
(d) 5V RMS:7.78nm  (e) 7V RMS:4.94nm  (f) 9V RMS:19.5nm

Fig. 9. Surface morphology change of C-face (0 0 0 –1) during anodizing in NaOH solutions observed by AFM.

(a) 0V RMS:0.286nm  (b) 2.5V RMS:0.682nm  (c) 3.5V RMS:1.36nm
(d) 5V RMS:6.27nm  (e) 7V RMS:10.5nm  (f) 9V RMS:12.3nm
could still be found on the oxidized surface in NaOH, demonstrating that the oxidation was not complete and the remaining material of the surface was still part of the original single crystal. On the other hand, oxides SiO$_x$ of different binding energy were observed on the SiC surface in both NaNO$_3$ and NaOH, which were probably the reason causing the differences in the surface morphology after oxidation. In the case of HNO$_3$, however, no oxide peak appeared, and a high peak of Si-C bond was found, which indicated that the surface was little oxidized. Instead, a peak of Si$_3$N$_4$ was separated. It was considered that the corrosion reaction of SiC in HNO$_3$ solution occurred, resulting in the silicon nitride. In general, Si-C bond appeared on the SiC surface after oxidation in both acidic and alkaline solutions, implying an incomplete oxidation process. Neutral electrolyte is the best choice to achieve a high degree oxidation process.

3.3. Oxidation rate in different electrolytes

3.3.1. Effect of polarization potential

The factor of oxidation rate is of significance in electrochemical-assisted hybrid machining process. In this study, the oxidation rates of 4H-SiC in five different solutions with 20 wt.% concentration were investigated based on the resulted oxidation film thickness and the polarization time. The results are shown in Fig. 12. It can be told from the figure that both the oxide rate and the oxygen content increased accordingly with increasing the polarization potential regardless of the electrolyte type. A high oxidation rates up to several nm/s could be achieved under 9 V. In addition, the curve of oxidation rate and oxygen content showed a high consistency. In comparison of different electrolytes, however, the oxide rate and oxygen content of 4H-SiC showed
a considerable difference, especially when the applied voltage was higher than 5 V. Specifically, the oxidizing ability of different electrolytes is NaCl (aq.) > NaNO₃ (aq.) > NaOH (aq.) > HNO₃ (aq.) = HCl (aq.), from strong to weak. By setting the applied potential, the oxidation rate and oxidation degree of 4H-SiC can be precisely controlled to achieve the required oxide film, which is of significant importance for controlling the process in the electrochemical-assisted hybrid machining.

3.3.2. Effect of electrolyte molar concentration

In previous study, electrolyte solutions of the same mass concentration were used, which caused difference in the molar concentration of ion. In this section, the anodization experiments were carried out with electrolytes of the same molar concentration. Fig. 13 shows the experimental results of oxidation film thickness and the oxidation rate in the initial 15 s. It was found that 4H-SiC could be electrochemically oxidized even at an extremely low molar concentration of 2 × 10⁻⁴ mol/L, whether in acidic, alkaline or neutral solution. The oxidation rate increased with increasing the molar concentration until it became saturated at mass concentration above 1 wt.%. It is considered that the reactive ion diffusion process determined the oxidation process when the molar concentration was low. The diffusion rate of ions increased with increasing the concentration before 1 wt.% resulting in a stronger oxidation reaction. On the other hand, when the molar concentration exceeded the critical value, in this case 1 mol/L, the oxidation rate did not increase anymore, and the oxidation process was dominated by electrical parameters at the high molar concentrations. In conclusion, the mass concentration of 1–2% enables optimal oxidation effect, with the largest film thickness of 197 nm and initial oxidation rate of 4.9 nm/s. It should be noticed that at above a certain value of high concentration, the oxidation rate decreased instead, which was considered due to the excessive current density resulted from the high molar concentration. It is well known that sufficiently high current density can break passive oxide film and cause transpassive dissolutions on oxidized surface [17]. This will be further discussed in the following section.

4. Discussion on the oxidation mechanism of 4H-SiC

Based on this study, it is considered that during the anodization, OH⁻ radical was generated at the anode surface by electrolyzing H₂O [18]. The chemically-unstable OH⁻ radical was a strong oxidizing active ion, which diffused from the bulk to the oxide-SiC interface and oxidized SiC to generate SiO₂ and CO/CO₂, as illustrated in Fig. 14. In an alkaline solution however, the SiO₂ film re-reacted with the OH⁻ to form SiO₃²⁻, thereby reducing the oxidation degree of the surface [14]. Similarly, the generated SiO₂ could also react with the acidic substance [19], which could refer to the dissolution of SiO₂ in a HF solution [15]. Therefore, the degree of oxidation in the alkaline and acidic solutions could not reach the neutral solution. However, the transpassive dissolution reaction was weak at a very low concentration, which was the reason why acidic and alkaline solution shows a high oxidation degree at low concentrations.

The ion in the neutral solution also plays an important role in
oxidation process. In NaCl, the chloride ion Cl\(^{-}\) possesses high electrochemical activity with strong penetrability in electrochemical reactions [20]. Therefore, it could enhance the ion diffusion process through the oxide layer and improve the oxidation process. In the case of NaNO\(_3\), however, since the reaction activity of NO\(_3\)\(^{-}\) was not high, ions mainly accumulated on the surface of the oxide film, resulting in a low reaction intensity and reaction rate.

Based on previous research [16] and the analysis above, we proposed the new reaction equations concerning the anodic oxidation occurring on the SiC surface as follows:

Anode:
\[
\begin{align*}
\text{OH}^- + h^+ & \rightarrow \text{OH} \\
2\text{OH}^- & \rightarrow 2\text{H}_2\text{O} + \text{O}_2 \\
\text{SiO}_2 + 4\text{OH}^- + 4h^+ & \rightarrow 2\text{SiO} + 4\text{H}_2\text{O} + 2\text{CO}_2 \\
\text{SiC} + 8\text{OH}^- + 8h^+ & \rightarrow \text{SiO}_2 + 4\text{H}_2\text{O} + \text{CO}_2 \\
\text{SiC} + 4\text{OH}^- + 4h^+ & \rightarrow \text{SiO} + 2\text{H}_2\text{O} + \text{CO}
\end{align*}
\]

5. Conclusions

The electrochemical oxidation characteristics of 4H-SiC in aspects of oxidation rate, oxidation film thickness and surface morphology etc. were experimentally investigated considering electrolyte types, ionic concentration and crystalline anisotropy using a three-electrode system. It was demonstrated that 4H-SiC could be easily oxidized even in an extremely low concentration electrolyte. Specifically, electrolyte of mass concentration 1–2% could achieve the optimal oxidation rate and oxidation film thickness. In addition, neutral electrolytes showed better oxidation ability than alkaline or acidic electrolytes. A maximum oxidation rate of 4.9 nm/s was successfully achieved by utilizing NaCl electrolyte solution with concentration 1 wt% and the resulted oxide film thickness was 197 nm. On the other hand, it was found that the oxidation rate of C-face was 5–15 times higher than that of Si-face depending on the applied voltage. The surface hardness of 4H-SiC could be reduced by 10–14 times after oxidation, which could significantly facilitate the mechanical removal process in hybrid machining. Based on the study, the mechanism of SiC oxidation in different electrolytes was discussed and the free radical OH\(^{•}\) was considered the main factor determining the oxidation of SiC. This study first gave a comprehensive investigation and the results have reference significance for improving the oxidation process in electrochemical-assisted hybrid machining of 4H-SiC or other similar material.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was partially supported and financial assistance by the National Natural Science Foundation of China (NSFC) (Project No. 51905255), Shenzhen Knowledge Innovation Plan (No. JCYJ20180504165815601), and Shenzhen High-level Innovation and Entrepreneurship Fund (No. KQTD20170810110250357).

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