Dry etching of silicon carbide in ICP with high anisotropy and etching rate

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Abstract. A detailed study of the influence of technological parameters of the plasma chemical etching process in inductively coupled plasma on the etching rate of single-crystal silicon carbide is presented. The physicochemical substantiation of experimentally revealed patterns is given. The optimal gas mixture was determined in terms of the etching rate of SiC. It was experimentally established that the dependence of the etching rate of silicon carbide on the percentage of oxygen in the total gas mixture is non-linear. Thus, with an increase in the percentage of O2 up to 23%, the etching rate of SiC gradually increases to 560 nm/min, a further increase in the percentage of O2 leads to a sharp decrease in the etching rate of SiC up to 160 nm/min at an oxygen content of 31%. The effect of the distance between the sample and the plasma generation zone on the etching rate of SiC was studied. It was shown that the greatest increase in speed is caused by an increase in the bias voltage, so at $U_{\text{bias}} = -50$ V the etching rate is 300 nm/min, and at $U_{\text{bias}} = -150$ V the value of the etching rate is 840 nm/min. The optimal parameters of the plasma-chemical etching process were selected for high-speed directional etching of single-crystal silicon carbide substrates.

1. Introduction

Silicon carbide is a wide-gap semiconductor material (the band gap lies in the range from 2.36 to 3.3 eV, depending on the crystal modification). The thermal conductivity of SiC under normal conditions is close to the thermal conductivity of copper, which ensures its use for heat removal in devices operating at high current densities. High thermal, radiation, and chemical resistance of silicon carbide is due to the high binding energy between Si and C, which ensures the stability of SiC-based devices in extreme operating conditions [1-6]. The large band gap allows the use of devices based on SiC in a very wide temperature range (up to 1000 °C). Due to the large values of the breakdown voltage, large specific powers are achieved, and high values of thermal conductivity simplify heat removal. Due to these properties, silicon carbide is a promising material for creating high-power high-frequency electronic devices and is also used as substrates for the epitaxial growth of AlIBV semiconductor materials (for example, gallium nitride) [1, 7-9].

The use of silicon carbide substrates imposes special requirements on the used technological processes and equipment. Today, the most promising technology for silicon carbide etching is plasma chemical etching (PCE) in inductively coupled plasma (ICP) [10-11]. The tasks of creating through
holes in silicon carbide plates, which are required when creating a number of devices and products of microsystem technology, are particularly relevant [10-13].

In this regard, the purpose of this work was to study the physicochemical laws of the process of plasma chemical etching of single-crystal silicon carbide in inductively coupled plasma.

2. Experimental technique
The experiments were carried out on a specially created original plasma chemical etching unit with an inductively coupled plasma source (figure 1).

![Figure 1. Simplified reactor layout (left) and 3 D model of PCE installation (right): 1 – discharge chamber; 2 – reaction chamber; 3 – loading and unloading flange of working samples; 4 – vacuum pumping system flange joint; 5 – heated substrate holder; 6 – cooled inductor; 7 – working gas mixture inlet; 8 – Faraday shield; 9 – inductor electromagnetic shield.]

For etching, samples of a 100 μm thick silicon carbide 6H-SiC were used. Before the PCE process, silicon carbide substrates were processed in argon (Ar) plasma for 10 minutes at the following parameters: ICP power (WRF) 750 W, Ubias = -35 V, reactor pressure (P) 0.75 Pa, argon flow rate (Q) 21.75 sccm, the distance between the sample and the plasma generation zone (h) is 15 cm. This operation was performed to remove unwanted contaminants from the surface of the SiC and the internal surfaces of the chamber rig.

SF₆ of high purity 99.998 (GOST TU 6-02-1249-83) was selected as the main gas for SiC etching. O₂ of a special purity was used as an additive to SF₆ (TU 2114-001-05798345-2007).

To evaluate the etching rate of the profile of the resulting structures and the etching depth after PCE, a CarlZeiss Supra 55VP scanning electron microscope (SEM) with an accuracy of ± 2.5% was used.

3. Results and discussions
It is known [14] that the dependence of the etching rate of single-crystal silicon carbide on the percentage of oxygen in the total gas etching mixture is complex and therefore requires special study. In experiments aimed at determining the dependence of the etching rate of SiC on the percentage of oxygen in the SF₆/O₂ gas mixture, the range of changes in the O₂ content varied from 9% to 31%, other technological parameters were recorded (table 1).
Table 1. The values of the technological parameters of experiments to study the dependence of the etching rate of SiC on the percentage of O₂ ([Q_{O₂}/ (Q_{O₂} + Q_{SF₆})] · 100%).

| W_{RF}, W | U_{bias}, V | P, Pa | h, sm | Q, % |
|-----------|------------|-------|-------|------|
| 800       | -100       | 0.75  | 5     | 9    |
| 800       | -100       | 0.75  | 5     | 16   |
| 800       | -100       | 0.75  | 5     | 23   |
| 800       | -100       | 0.75  | 5     | 28   |
| 800       | -100       | 0.75  | 5     | 31   |

Figure 2. Dependence of the etching rate of SiC on the percentage of oxygen in the etching mixture.

It can be seen from the graph (figure 2), the etching rate of SiC gradually increases (up to 560 nm/min) with an increase in the percentage of oxygen up to 23%, most likely due to an increase in the concentration of F * radicals [15] and the intensification of the formation of volatile compounds, CO, CO₂, COF₂. With a further increase in oxygen concentration, a significant decrease in the etching rate (up to 160 nm/min) occurs due to a decrease in the concentration (due to over-dilution) of the etchant gas and the possible formation of silicon oxide on the surface of the treated surface, which prevents SiC etching [16].

Table 2. The values of the technological parameters of experiments to study the dependence of the etching rate of SiC on the bias voltage.

| W_{RF}, W | U_{bias}, V | P, Pa | h, sm | Q, % |
|-----------|------------|-------|-------|------|
| 800       | -50        | 0.75  | 5     | 23   |
| 800       | -75        | 0.75  | 5     | 23   |
| 800       | -100       | 0.75  | 5     | 23   |
| 800       | -125       | 0.75  | 5     | 23   |
| 800       | -150       | 0.75  | 5     | 23   |
An increase in the bias potential on the substrate makes it possible to significantly increase the etching rate of various materials at constant values of the remaining technological parameters [12, 17, 18].

An increase in the bias voltage applied to the substrate leads to a growth of energy ions directed to the etching surface, which increases the intensity of the sputtering process. Table 2 shows the experimental conditions; the etching time in all experiments was 30 minutes.

It can be seen from the data in figure 3, an increase in the bias voltage leads to a significant (2.8-fold) increase in the etching rate (up to 840 nm/min), and the obtained dependence is almost linear.

![Figure 3. Dependence of the etching rate on the bias voltage applied to the substrate holder.](image)

The influence of the distance between the sample and the plasma generation zone is poorly studied. However, this dependence is of interest from the point of view of increasing the efficiency of the SiC PCE process. In order to study the nature of the influence of this parameter on the etching rate, a special series of experiments was carried out, the conditions for which are shown in table 3.

| W<sub>RF</sub>, W | U<sub>bias</sub>, V | P, Pa | h, sm | Q, % |
|------------------|------------------|------|-------|-----|
| 800              | -150             | 0.75 | 5     | 23  |
| 800              | -150             | 0.75 | 7.5   | 23  |
| 800              | -150             | 0.75 | 10    | 23  |
| 800              | -150             | 0.75 | 12.5  | 23  |
| 800              | -150             | 0.75 | 15    | 23  |

As can be seen from the graph (figure 4), the dependence of the etching rate of SiC on h is linear. As the substrate approaches the plasma zone from 15 cm to 5 cm, the velocity increases linearly from 0.72 μm/min to only 0.84 μm/min, indicating that this dependence is relatively weak.
Figure 4. Dependence of the etching rate of SiC on the distance between the substrate of the plasma generation zone.

The increase in the SiC etching rate with the sample approaching the plasma generation zone is likely due to the presence of an ion gradient in the direction from the discharge chamber to the substrate holder. As the etching surface approaches the plasma generation unit, the number of ions reaching the surface of the material grows, which is the reason for the increase in the etching rate.

An analysis of the results enables to establish the technological parameters inherent in the technological equipment used to create high-speed directional plasma-chemical etching of SiC.

Table 4. The values of the technological parameters of the control experiment of the high-speed directed process of PCE SiC.

| $W_{RF}, W$ | $U_{bias}, V$ | $P, Pa$ | $h, sm$ | $Q, \%$ | $t, min$ |
|-------------|-------------|--------|--------|--------|--------|
| 800         | -200        | 0.75   | 5      | 23     | 30     |

As a sample, 6H-SiC with a thickness of 350 μm was used, with a 1-μm thick chromium (Cr) mask, and etching windows of various geometries. Technological parameters of the control experiment are presented in table 4.

Figure 5. Microphotographs of directional structures in SiC in a control experiment.
The results of a control experiment of high-speed directional etching of single-crystal silicon carbide are presented in figure 5.

To increase the etching rate, it was also decided to increase the bias voltage to \(-200\) V. The etching rate of SiC was about \(0.93\) \(\mu\)m/min with the inclination angle of the etching window profile wall of \(90^\circ\).

4. Conclusions
Comparing the obtained results with the literature data on the plasma-chemical etching of silicon carbide, it should be noted that in the vast majority of works the achieved etching rates lie in the range from 200 to 700 nm/min [10, 14, 19, 20–24] at the RF power of the ICP source less than 1000 W and a bias voltage of less than \(-300\) V and from 700 nm/min to 1500 nm/min at a RF power of the source of more than 1000 W and a bias voltage of more than 300 V [15, 25]. It is especially worth noting the authors of [26, 27] who managed to achieve record etching rates of silicon carbide 4H-SiC substrates (of the order of 2 \(\mu\)m/min) at etching depths of more than 140 \(\mu\)m, however, these values were achieved at an RF power of 2 kW and voltage displacements of more than 500 V. Noteworthy are the results obtained in [28], the authors of which report that the etching rate of silicon carbide 4H-SiC substrates reaches about 0.95 \(\mu\)m/min with an etching depth of 330 \(\mu\)m and the absorbed power and bias voltage of 1200 W, -160 V, respectively. It was possible to achieve such speeds through the use of a rather complex helicon source of high-density plasma.

Etching rates of 0.93 \(\mu\)m/min with an etching depth of about 28 \(\mu\)m with an RF source power of 800 W and a bias voltage of -200 V with the inclination angle of the etching window profile wall of 90° have been achieved.

References
[1] Zhe C F 2003 *Silicon Carbide: Materials, Processing & Device* (CRC Press) p 416
[2] Tschumak E 2009 Comparative study of 3C-GaN grown on semi-insulating 3C-SiC/Si (100) substrates *Materials Science Forum* 615-17 943-6
[3] Severino A 2011 3C-SiC film growth on Si substrates *ECS Transactions* 35(6) 99-116
[4] Kim K C 2001 Formation mechanism of interfacial voids in the growth of SiC films on Si *J. of Vacuum Science & Technology A: Vacuum, Surfaces, and Films* 19(5) 2636-41
[5] Lebedev A A 2006 Heterojunctions and superlattices based on silicon carbide *Semiconductor science and technology* 21(6) 17-34
[6] Zetterling C M 2002 *Process technology for silicon carbide devices* (IET) 2
[7] Wang C, Lee W S, Cho S J and Kim N Y 2012 SiC backside source grounding process for AlGaN/GaN HEMT by physical dicing method *Electronic Letters* 48(7) 405–6
[8] Cho S J, Wang C and Kim N Y 2012 Effects of double passivation for optimize DC properties in gamma-gate AlGaN/GaN high electron mobility transistor by plasma enhanced chemical vapor deposition *Thin Solid Films* 520(13) 4455
[9] Zhou Q et al 2011 GaN/SiC avalanche photodiodes *Appl. Phys. Lett.* 99 131110
[10] Osipov K Y and Velikovskiy L E 2012 Formation technology of through metallized holes to sources of high-power GaN/SiC high electron mobility transistors *Semiconductors* 46(9) 1216-20
[11] Osipov A A et al 2018 Etching of SiC in Low Power Inductively-Coupled Plasma *Russian Microelectronics* 47(6) 427-33
[12] Ekinci H 2015 Plasma etching of n-Type 4H-SiC for photoconductive semiconductor switch applications *J. of Electronic Materials* 44(5) 1300-5
[13] Voss L F 2008 SiC via fabrication for wide-band-gap high electron mobility transistor/microwave monolithic integrated circuit *J. of Vacuum Science & Technology B: Microelectronics and Nanometer Structures Processing, Measurement, and Phenomena* 26(2) 487-94
[14] Jiang L 2003 Inductively coupled plasma etching of SiC in SF\(_6\)/O\(_2\) and etch-induced surface chemical bonding modifications *J.of Applied Physics* 93(3) 1376-83
[15] Khan F A 1999 High rate etching of SiC using inductively coupled plasma reactive ion etching in SF$_6$-based gas mixtures *Applied physics letters* **75**(15) 2268-70
[16] Pan W S 1990 Reactive ion etching of SiC thin films by mixtures of fluorinated gases *J. of the Electrochemical Society* **137**(1) 212-20
[17] Plank N O V 2003 The etching of silicon carbide in inductively coupled SF$_6$/O$_2$ plasma *J. of Physics D: Applied Physics* **36**(5) 482-7
[18] Kim D W 2004 High rate etching of 6H–SiC in SF$_6$-based magnetically-enhanced inductively coupled plasmas *Thin Solid Films* **447** 100-4
[19] Choi J H 2012 Fabrication of SiC nanopillars by inductively coupled SF$_6$/O$_2$ plasma *Materials Science Forum* **711** 66-9
[20] Cho H 2000 Ultradeep, low-damage dry etching of SiC *Applied Physics Letters* **76**(6) 739-41
[21] Beheim G 2002 *Deep reactive ion etching for bulk micromachining of silicon carbide* (CRC Press LLC:USA) pp 21-1-21-12
[22] Camara N 2002 Study of the reactive ion etching of 6H–SiC and 4H–SiC in SF$_6$/Ar plasmas by optical emission spectroscopy and laser interferometry *Solid-State Electronics* **46**(11) 1959-63
[23] Ahn S C 2004 A study on the reactive ion etching of SiC single crystals using inductively coupled plasma of SF$_6$-based gas mixtures *Metals and Materials International* **10**(1) 103-6
[24] Jiang L 2004 Dry etching of SiC in inductively coupled Cl$_2$/Ar plasma *J. of Physics D: Applied Physics* **37**(13) 1809-14
[25] Ruan J A Low 2010 RF power SiC substrate via etch Int.Conf. on Compound Semiconductor Manufacturing Technology CS MANTECH Conf. (Portland Oregon USA)
[26] Okamoto N 2009 SiC backside via-hole process for GaN HEMT MMICs using high etch rate ICP etching CS MANTECH Conf. (Tampa Florida USA)
[27] Okamoto N 2009 Differential etching behavior between semi-insulating and n-doped 4H-SiC in high-density SF$_6$/O$_2$ inductively coupled plasma *J. of Vacuum Science & Technology A: Vacuum, Surfaces, and Films* **27**(3) 456-60
[28] Chabert P Deep etching of silicon carbide for micromachining applications: Etch rates and etch mechanisms 2001 *J.of Vacuum Science & Technology B: Microelectronics and Nanometer Structures Processing, Measurement, and Phenomena* **19**(4) 1339-45