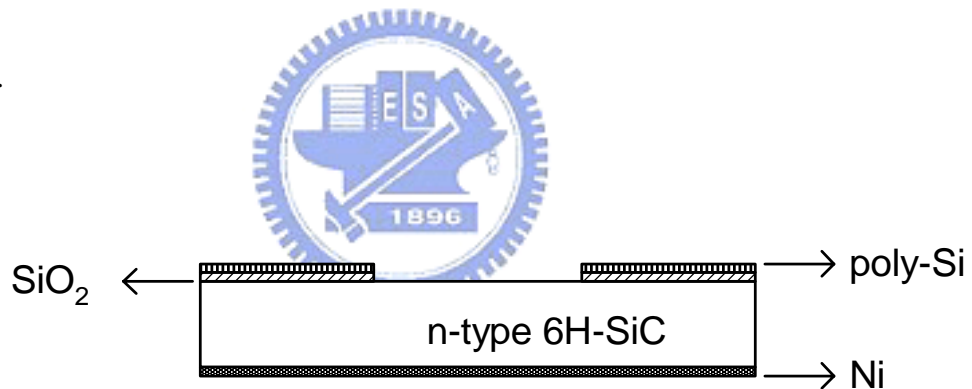


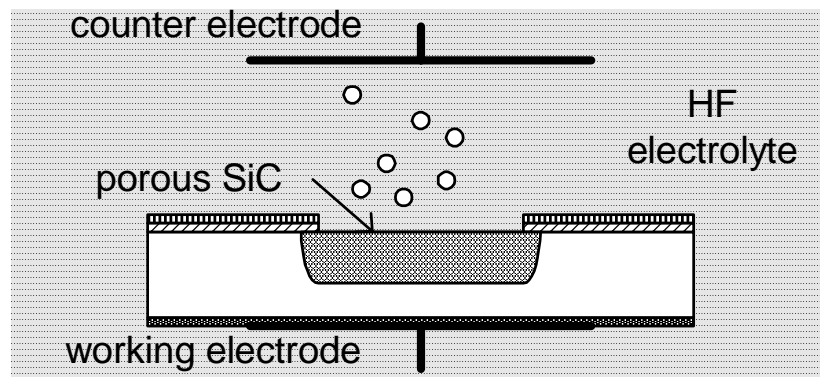
APPENDIX A

N-TYPE 6H-SiC ETCHING PROCESS

The whole etching process is illustrated in Fig. A.1. After the preparation of the n-type 6H-SiC sample (Fig. A.1 (a)), it is anodized in an HF electrolyte (Fig. A.1 (b)). During the anodization process, a porous layer is formed. This layer can be oxidized easily. After a wet thermal oxidation of the sample (Fig. A.1 (c)), the thick oxidized porous SiC layer is removed in a concentrated HF solution (Fig. A.1 (d)). In this way, a deep cavity is obtained. In the following, the different process steps are discussed in more detail.



(a) sample with masking layers and back-side contacts



(b) anodization

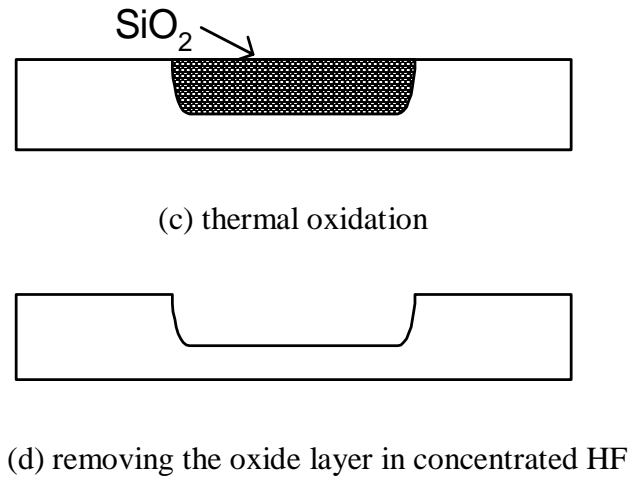


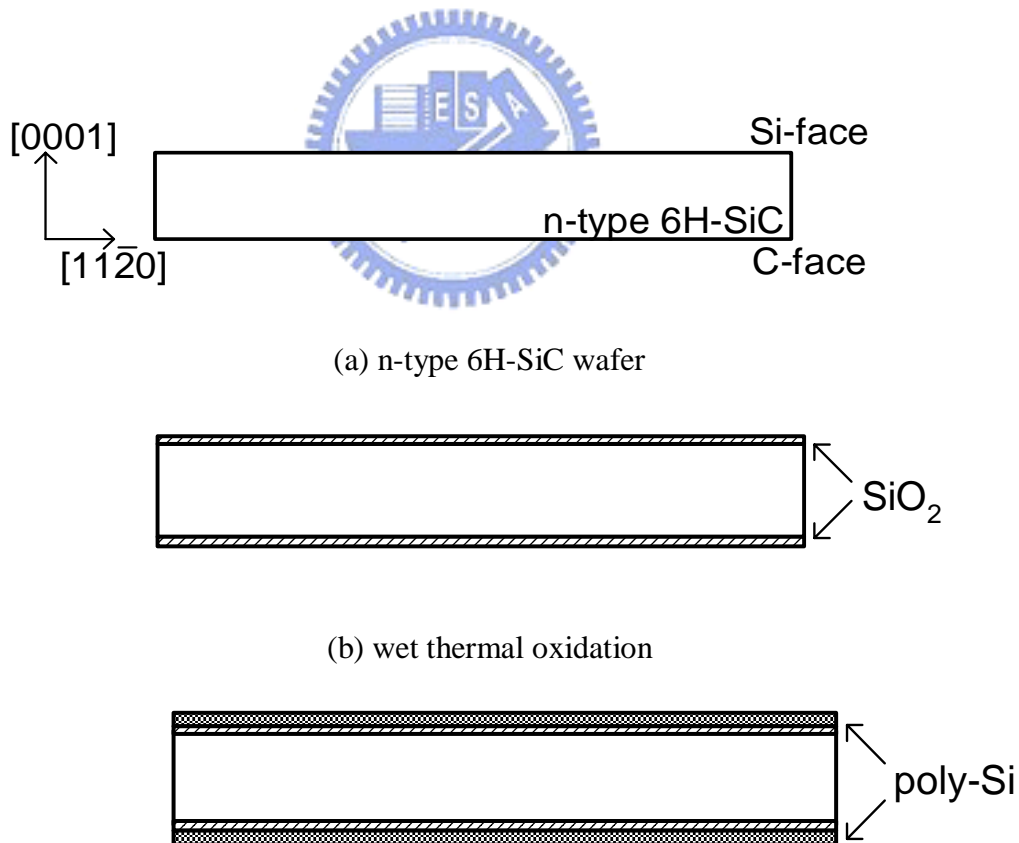
Figure A.1. Main process steps of the etching process for n-type 6H-SiC.

A.1 Sample Preparation

Before the thick porous layer formation in an HF electrolyte, a passivation layer has to be found, which is suitable for a long-term anodization as well as for the preparation of an ohmic contact to the n-type 6H-SiC sample. A mask scheme with a 600 nm LPCVD (Low Pressure Chemical Vapor Deposition) poly-Si layer on the top of the oxide layer of the 6H-SiC sample is used because this mask can sustain the attack from HF solution making electrochemical etching of SiC possible. Nickel is well suited for ohmic contacts to n-type SiC and is therefore used in our experiment as contact metal.

The preparation process of the n-type 6H-SiC samples is illustrated in Fig. A.2. For this process, single crystal n-type 6H-SiC wafers with a doping concentration

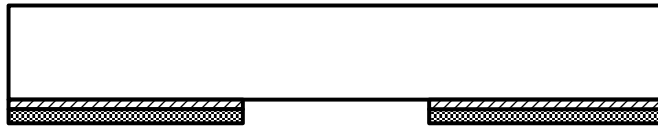
between $1-4 \times 10^{17} \text{ cm}^{-3}$ from SiCrystal AG, Germany, are used. After RCA cleaning, wet thermal oxidation (Fig. A.2 (b)) and LPCVD of poly-crystalline silicon (Fig. A.2 (c)) are performed to create the etching mask for the anodization process. The mask layer is deposited on the C-face of the 6H-SiC wafers and patterned using a standard photolithographic process (Fig. A.2 (d and e)). Finally, a 200 nm thick nickel layer is sputtered onto the opposite side of the wafer by a Magetron sputter system and annealed at 1000°C to obtain an ohmic contact (Fig. A.2 (f)).



(c) LPCVD poly-Si



(d) poly-Si etching by RIE



(e) SiO₂ etching by HF solution



(f) Ni sputtering and annealing

Figure A.2. Preparation of n-type 6H-SiC samples to be etched.

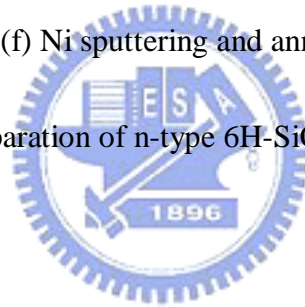


Fig. A.3 shows the setup used in the experiments. The HF electrolyte is kept in a Teflon etching tank. The SiC sample, which serves as the working electrode, is attached to a holder and sealed by wax to protect the backside of the sample. A platinum plate is used as the counter electrode. The working and counter electrodes are connected to a constant current source. During the etching process, the voltage and current are monitored by multimeters and recorded by a computer with a GPIB interface.

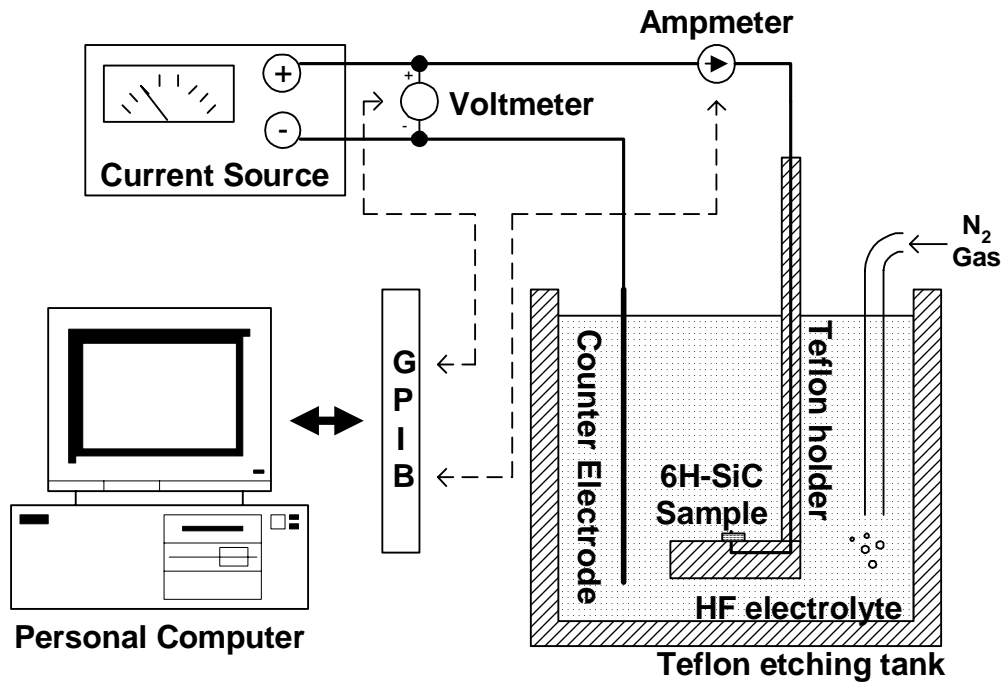


Figure A.3. Setup for the n-type 6H-SiC electrochemical etching.



A.2 Anodization Process

The prepared samples are then anodized in a 2% HF solution for one hour, using a current density of 50 mA/cm^2 and the change of the voltage during the anodization is recorded. After the anodization, the samples are cut with a dicing saw for examination. A typical cross-section is illustrated in Fig. A.4. As we can see, porous SiC layers are formed as a result of the anodization process. Three porous areas with different pore sizes can be identified. The anodized surface (area 1) has the finest pores whose diameter is about 20 nm, the intermediate area (area 2) consists of pores

with a diameter of about 100 nm. The thickest part of the porous structure (area 3) has pores with a diameter of about 1 μm and extends to the substrate.

Because the bond between areas 2 and 3 is not so strong, area 1 and 2 are easily released during the anodization. The pore walls in area 3 are important. If the pore walls are too thick or non-uniform, they cannot be fully oxidized because the thermal oxidation rate of SiC is quite low in comparison with silicon. As illustrated in Fig. A.5, with an 8% HF electrolyte, the anodized area shows extremely non-uniform porous structure. In addition, the profiles of the lateral and vertical anodized areas are also irregular. Such results make not only the thermal oxidation difficult but also the profile irregular. It is experimentally determined that the use of a 2% HF electrolyte results in a pore structure with superior uniformity and thickness compared to samples etched with a higher concentration of HF. Moreover, the use of a 2% HF solution does not require high voltages as it is the case for lower HF concentration.

The optimum value for the current density, used for the SiC anodization is determined in the same way. With a higher current density, a stronger reaction will occur and generate a large number of bubbles, which will result in a poor porous structure. With a lower current density, a more uniform porous structure can be achieved. However, the anodization rate is lower in this case. Finally, a current density of 50 mA/cm^2 is recommended as the optimum value for the process.

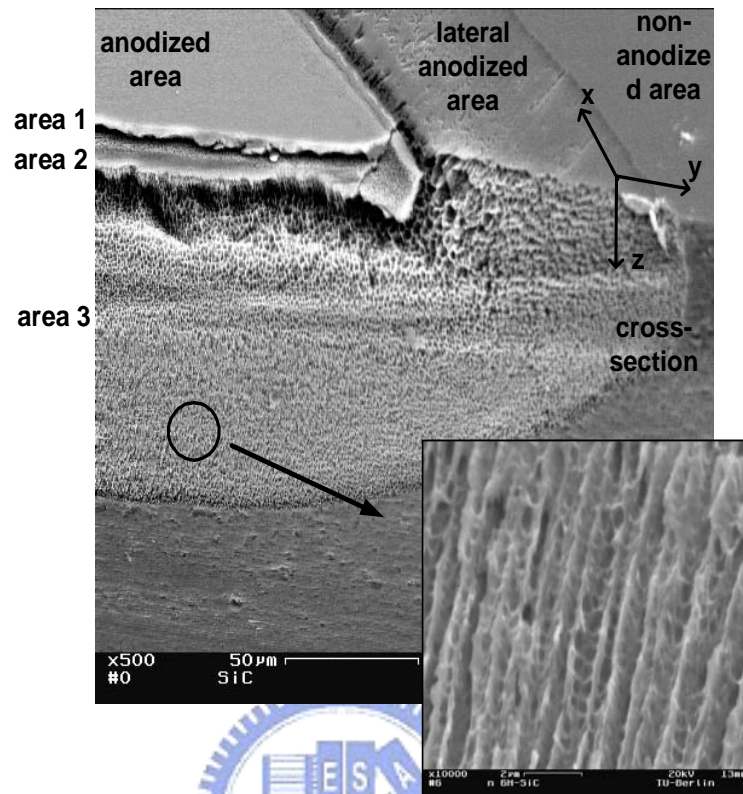


Figure A.4. Cross-section of an n-type 6H-SiC sample after 2 hour anodization (50mA/cm², 2% HF).

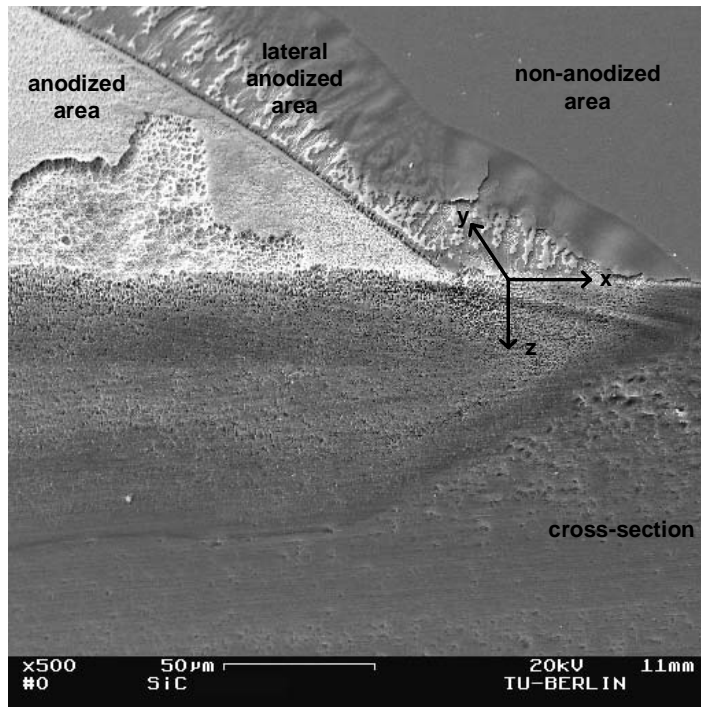


Figure A.5. Cross-section of an n-type 6H-SiC sample with a higher HF concentration.

A.3 Oxidation Process

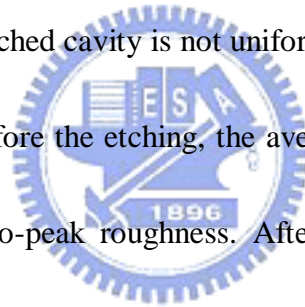


After the anodization process, the samples are oxidized in a wet thermal oxidation environment at a temperature of 1150°C. After 10 hours, the samples are removed and cut by a dicing saw as previously described. As illustrated in Fig. A.6, the pore walls in area 3 are thicker and thus make the pores smaller after the oxidation.

Then, the oxidized samples are immersed in a concentrated HF to remove the thick porous oxide layer. After removing the oxidized porous layer, the porous structure can still be seen at the bottom of the cavities, as shown in Fig. A.7. The

remaining pores have thicker pore walls, which make them difficult to remove, even after a 24 hours oxidation. The depth of the remaining pores is less than 1 μm . The etched depth with respect to the anodization time is illustrated in Fig. A.8. The relation shows a linear dependence, which means one can obtain the desired etching depth by adjusting the anodization time.

An n-type 6H-SiC circular cavity etched by the developed method is illustrated in Fig. A.9. A profile measured by a Tencor alpha-step profile meter is illustrated in Fig. A.10. An etched depth of about 150 μm is reached by time-controlled etching. However, the bottom of the etched cavity is not uniform. The roughness of the surface is illustrated in Fig. A.11. Before the etching, the average roughness is about 3.2 nm and has about 34 nm peak-to-peak roughness. After the etching, the average and peak-to-peak roughness are about 780 nm and 4 μm , respectively.



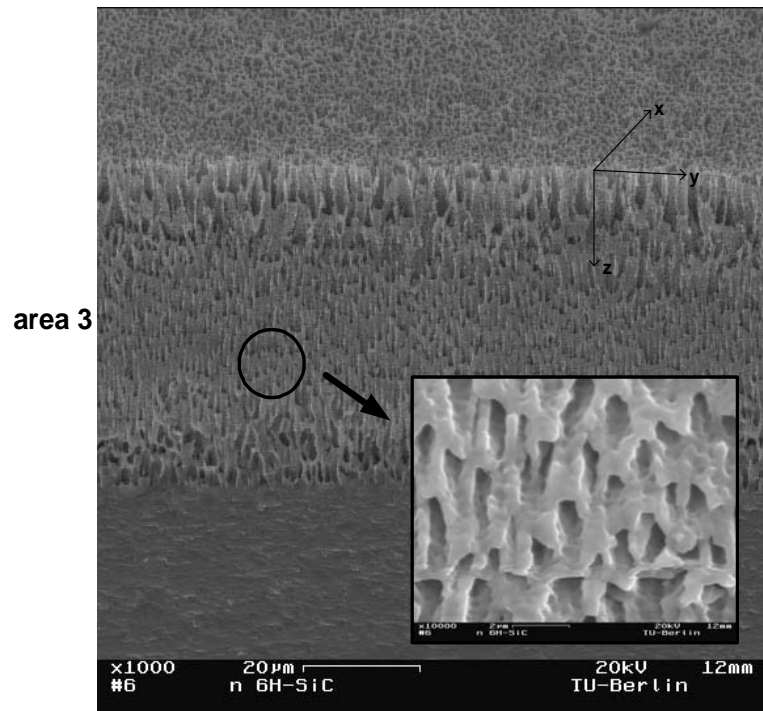


Figure A.6. Cross-section of the anodized n-type 6H-SiC sample after 10 hours wet thermal oxidation.



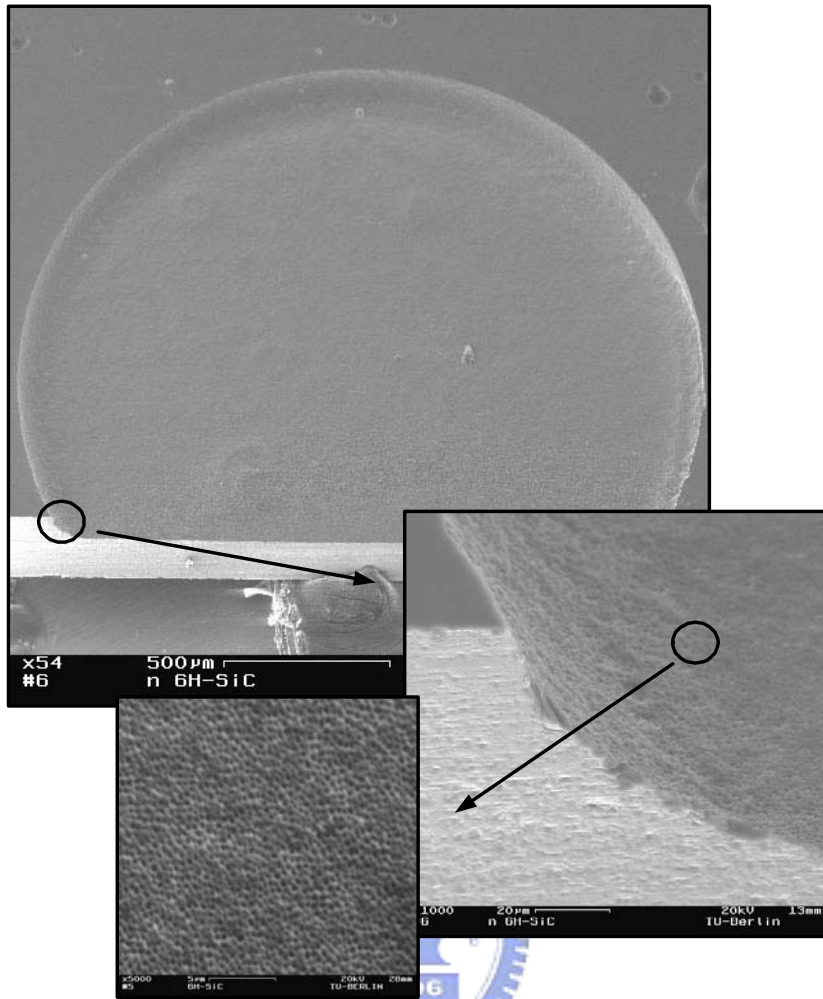


Figure A.7. Appearance of the porous structure after 10 hours oxidation of 1150°C.

The oxidized porous layer is then etched in an HF solution.

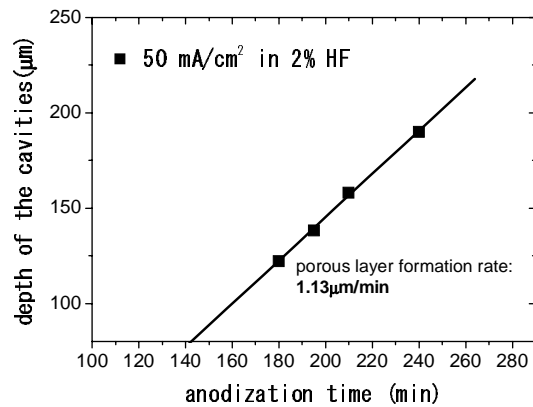


Figure A.8. Depth of the cavities V.S. anodization time.

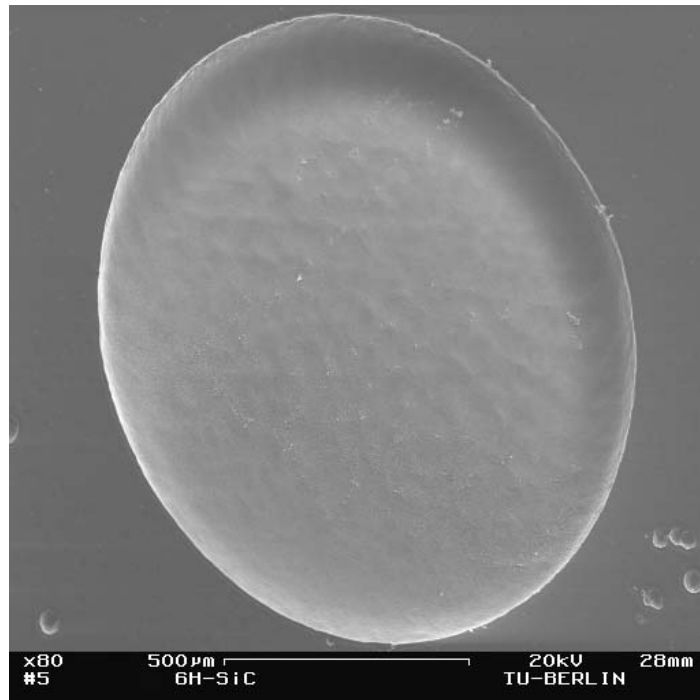


Figure A.9. Photograph of an etched 6H-SiC cavity.

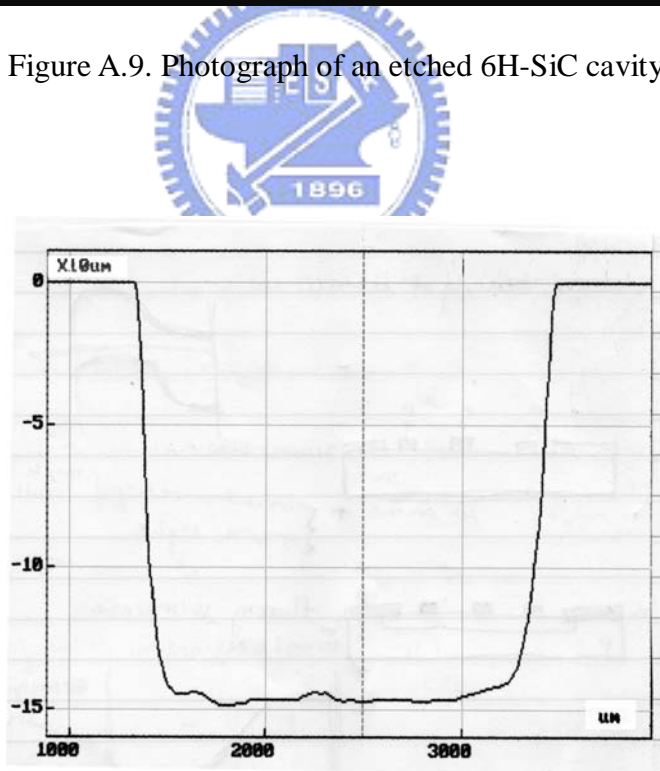


Figure A.10. Profile of the etched 6H-SiC cavity.

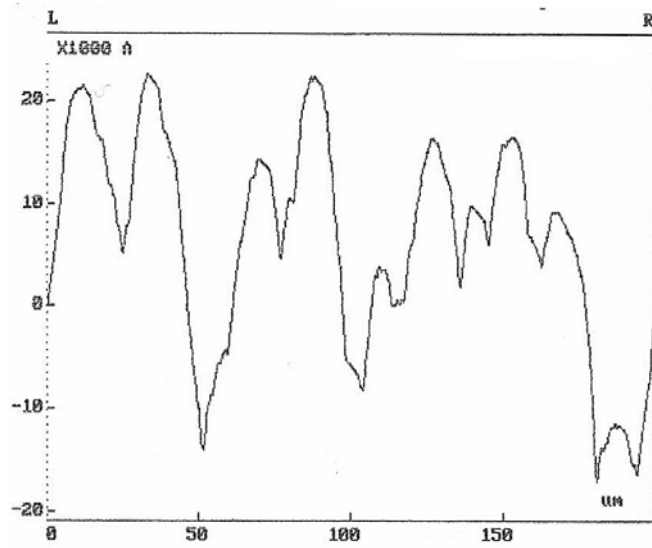


Figure A.11. Roughness of the n-type 6H-SiC surface after etching.

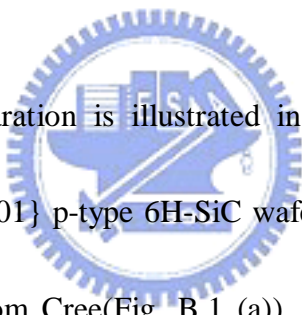


APPENDIX B

P-TYPE 6H-SiC ETCHING PROCESS

The etching process of p-type 6H-SiC is similar as the one of n-type 6H-SiC. The main difference is the ohmic contact, which is realized with nickel for n-type 6H-SiC and aluminum for p-type SiC. In the following paragraph, the different process steps are discussed in more detail.

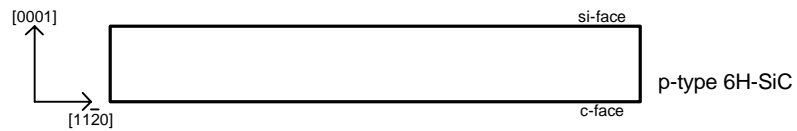
B.1 Sample Preparation



The whole sample preparation is illustrated in Fig. B.1 and is described as follows: 3.5° off-oriented {0001} p-type 6H-SiC wafers with a doping concentration of 2×10^{18} were purchased from Cree(Fig. B.1 (a)). After the modified RCA clean process, the oxide layer is grown at 1150°C for 5 hours(Fig. B.1 (b)). Then, an 890nm thick polysilicon layer is deposited at 590°C by LPCVD(Fig. B.1 (c)). The areas to be etched are opened on the C-face of the silicon carbide wafer, using a standard photolithography step(Fig. B.1 (d)). The polysilicon and oxide layer on the Si-face are removed by RIE and a HF dip respectively(Fig. B.1 (d) and (e)). Finally, a 1 μm thick Al/Si 99/01 is sputtered on the Si-face of 6H-SiC by magnetron sputtering(Fig. B.1 (f)). As-deposited, the contact between 6H-SiC and Al behaves like an open-circuit.

During annealing at 800°C for 6 minutes in an N₂ environment, an ohmic contact is formed and the current-voltage curve becomes linear.

The setup for the electrochemical etching used in our experiments is the same as the one that is used in n-type 6H-SiC electrochemical etching and is illustrated in APPENDIX A.



(a) p-type 6H-SiC substrate



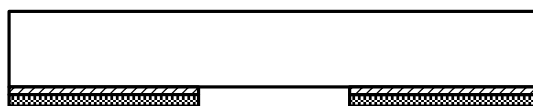
(b) wet thermal oxidation



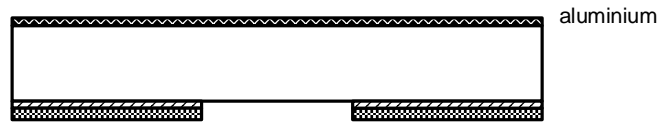
(c) deposition of LPCVD poly-Si



(d) patterning and removing poly-Si by RIE



(e) etching SiO₂ by HF



(f) sputtering and annealing Al

Figure B.1. Process flow for the preparation of p-type 6H-SiC samples.

B.2 Constant Current Electrochemical Etching

In order to obtain a linear relationship between the etching time and the etching depth, the samples are anodized at a constant current density. During the etching, the average working voltage remains constant and does not show any evident climb or drop during the anodization process. This means that the anodization process will continue as long as electrical charges are constantly supplied.

When an electrolyte with a lower concentration of HF is used, the working voltage increases at the same current density due to the low conductivity of the electrolyte. In our experiments, when a 1% HF was used as electrolyte, the voltage was over the voltage-limit protection of the current source at a current density of $100\text{mA}/\text{cm}^2$. When the corresponding voltage is higher than this limit, the supplied current will be reduced in order to protect the equipment. In this situation, the total transferred charged per time unit is not uniform, so the etching rate will also not be uniform. This situation must be avoided if a constant etching rate is desired.

B.3 Etching Profile

During the anodization, the p-type 6H-SiC begins to dissolve. The anodic etching process of SiC is believed to consist of two steps: first, electrochemical oxidation of silicon carbide occurs at the interface between the SiC sample and electrolyte. The as-formed oxidation layer is then removed by HF in the second step. The etching rates with different HF concentrations and current densities are shown in Table 1. There are some interesting points that we can find in Table 1. Firstly, the relation between etching depth and etching time at the same current density and electrolyte concentration is linear. With this linear dependence, we can obtain the desired etching depth by controlling the etching time. Secondly, the etching rate is proportional to the current density for the same HF concentration. This means that we can control the etching rate by the current density. However, a higher current density will result in a stronger reaction, which will introduce a large number of bubbles making the etched surface non uniform and rough. Thirdly, at the same current density, the etching rates are similar for HF concentrations of 4% and 8%. But it decreases for HF concentrations lower than 4%. This situation may indicate that a different number of charges is consumed or that other reactions are taking place, such as electrolysis of water, and consume the applied charges when a different concentration of electrolyte

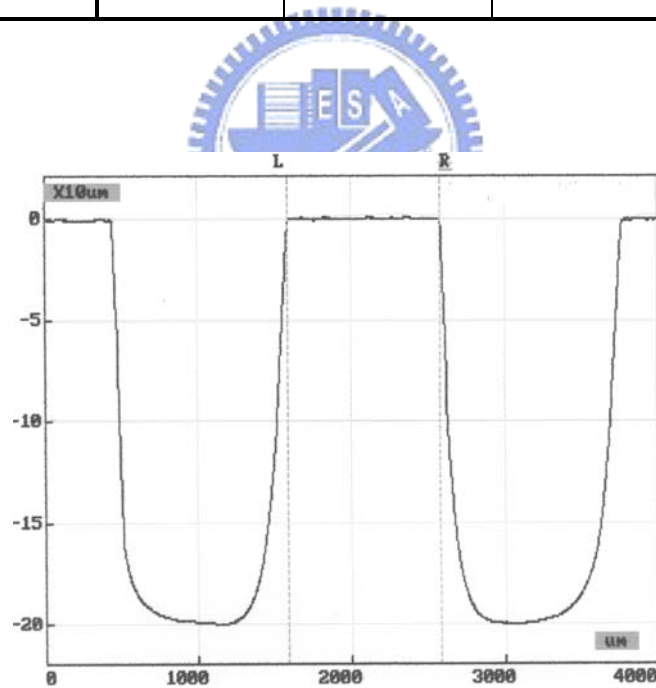
is used.

An etching profile obtained with 2% HF electrolyte and a $50\text{mA}/\text{cm}^2$ current density measured with a Tencor alpha-step profile meter is illustrated in Fig. B.2. In Fig. B.2 (a), an etching depth of $200\mu\text{m}$ is obtained by time-controlled etching. The average roughness of the surface of the p-type 6H-SiC sample before the etching, which is shown in Fig. B.2 (b), is 116nm , while the roughness of the etched surface is only 83nm after the etching (Fig. B.2 (c)). The etched profiles resulting from different etching conditions are examined by SEM (Scanning Electron Microscopy). Fig. B.3 demonstrates an etched p-type 6H-SiC substrate with a circular center boss structure, which could be used for applications like pressure sensors.

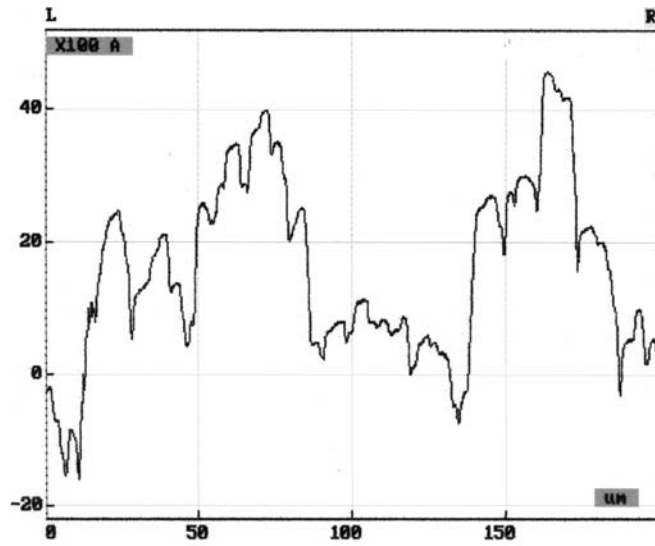


Table B.1. p-type 6H-SiC anodic etching rate (unit: $\mu\text{m}/\text{min}$) with respect to different HF concentration and current density

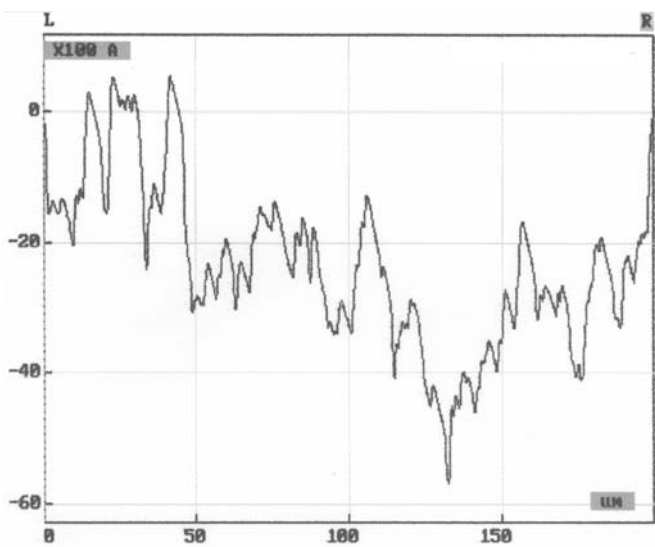
current density (mA/cm^2) \backslash HF	1%	2%	4%	8%
	50	0.24	0.40	0.49
100	0.43	0.67	0.95	0.96
150	--	0.88	1.31	1.48



(a)



(b)



(c)

Figure B.2. Profiles measured by a profile meter (a) an anodic etched annular structure with a 200 μm deep annular cavity (b) the roughness of the structure before anodic etching and (c) after anodic etching.

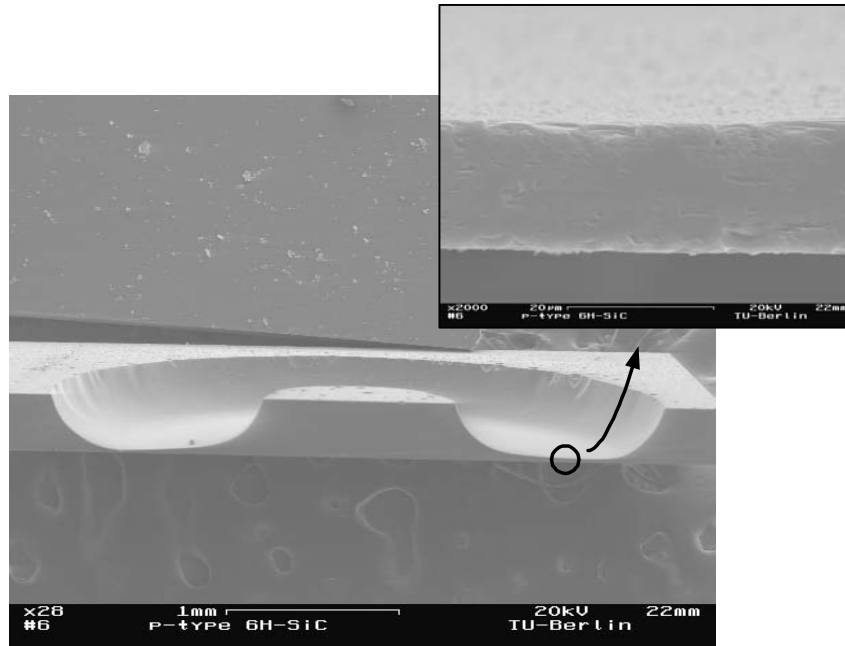


Figure B.3. An electrochemical etched p-type 6H-SiC annular structure. In the right-up window, the membrane about 20 μm thick is magnified from the annular structure.

