Electrochemical Etching of n-Type 6H-SiC Without UV Illumination

Wei-Hsu Chang, Bernt Schellin, Ernst Obermeier, *Member, IEEE*, and Yu-Chung Huang

***Abstract*—Deep etching of n-type 6H-SiC using a two-step etching process has been studied. First, anodization of 6H-SiC in an HF electrolyte (2 wt.%) without ultraviolet light is applied to form a deep porous layer with the desired dimensions. Then, a thermal oxidation process is used to oxidize this porous layer. The oxidized layer is then removed in a concentrated HF solution. In the experiments, the etching parameters electrolyte concentration and current density are optimized in order to obtain a uniform pore size and hence, a smooth etched surface. After adjusting these parameters, the porous layer formation experiments are carried out at 20 °C in a 2 wt.% HF electrolyte using a current density of 50 mA/cm^2. The corresponding porous layer formation rate is about 1.1 μm/min. To demonstrate the capabilities of this SiC bulk micromachining process, deep circular cavities are fabricated in n-type 6H-SiC substrates. [1134]**

***Index Terms*—Bulk micromachining, electrochemical etching, n-type 6H-SiC, porous 6H-SiC.**

I. Introduction

It is believed that SiC is a promising material for electronic devices operating at high temperatures, high power, and high frequency because it exhibits a larger bandgap, a higher breakdown field, a higher thermal conductivity, and a higher saturation velocity in comparison to Si [1]. It is also known for its mechanical hardness, chemical inertness, high thermal conductivity, and electrical stability, making it an excellent material for sensors and actuators for harsh environment applications [2]–[4].

Before one can use this material for microsystem applications, a suitable etching technology, or so-called bulk micromachining, has to be developed. So far, no wet etchants are known that have the ability to etch 6H-SiC at room temperature. However, a feasible way to etch SiC at room temperature is electrochemical etching [5]. It is known that positive charge carriers (holes) are required at the surface of the semiconductor in order to perform electrochemical etching. For n-type 6H-SiC, holes could be obtained either by photo induced electron-hole-pair generation or by an avalanche or Zener breakdown. Based on the photoanodic etching method, Shor [6] proposed a two-step method to etch SiC: i) anodization with ultraviolet (UV) illumination to form a porous SiC layer and ii) removal of the porous layer by oxidation and HF etching. Up to now, however, no one has demonstrated deep etching of n-type 6H-SiC with this method. In fact, the initial porous layer hinders the UV-light illumination of the material underneath it, thereby preventing further thickening of the porous layer. Konstantinov [7] compared samples prepared using UV light-assisted porous layer formation with those obtained without illumination. He showed that an n-type 6H-SiC porous layer can indeed be formed without UV light. From 1996 to 1998, Okojie [8], [9] made pressure sensors by etching n-type 6H-SiC without illumination. However, he did not report details about the etching procedure. In this paper, we present a feasible process for the bulk micromachining of n-type 6H-SiC.

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II. Experimental

Before starting the etching experiments, a suitable passivation layer for the long term anodization process in an HF electrolyte and an ohmic contact material for the backside contact of the n-SiC sample have to be found. A mask scheme with a low-pressure chemical vapor deposition (LPCVD) poly-Si layer on the top of an oxide layer 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 [16] and is therefore used as contact metal in our experiments.

The preparation process for the n-type 6H-SiC samples is illustrated in Fig. 1. For this process, single crystalline n-type 6H-SiC wafers with a doping concentration between 1–4$\times 10^{17}$ cm^{-3} from SiCrystal AG, Germany, are used. After RCA cleaning, the oxide layer is grown by wet thermal oxidation at 1150 °C for 3 h [Fig. 1(b)]. Then, 600 nm of LPCVD polysilicon [Fig. 1(c)] are deposited at 620 °C to form 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. 1(d) and (e)]. Finally, a 200-nm-thick nickel layer is sputtered onto the opposite side of the wafer by a magnetron sputter system and annealed at 1000 °C to obtain an ohmic contact [Fig. 1(f)].

The electrochemical etching process is illustrated in Fig. 2. The prepared n-type 6H-SiC samples [Fig. 2(a)] are anodized in an HF electrolyte [Fig. 2(b)], using a constant current density. During the anodization process, a porous SiC layer is formed in the area of the later cavity. This porous layer can be oxidized easily. After a wet thermal oxidation of the samples [Fig. 2(c)], the thick oxidized porous SiC layer is removed in a concentrated HF solution [Fig. 2(d)]. In this way, deep etched cavities are obtained.

The setup used in the experiments is described as follows. The HF electrolyte is kept in a Teflon etching tank. A SiC sample,

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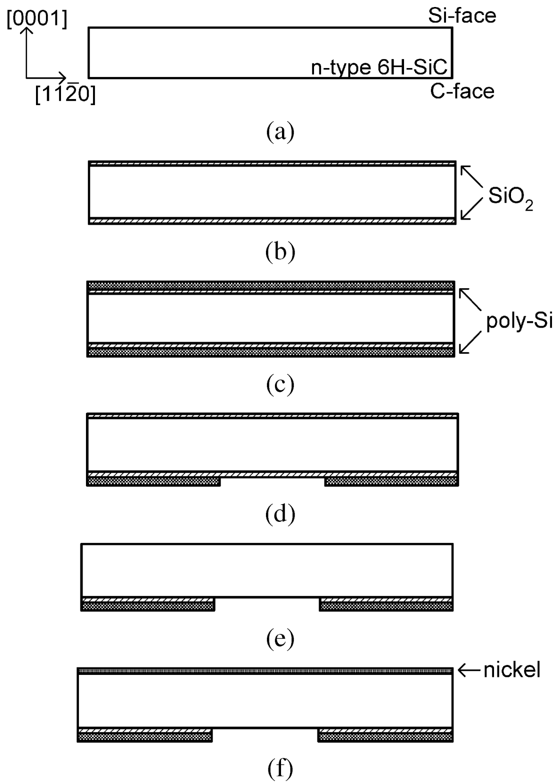

Fig. 1. Preparation of n-type 6H-SiC samples to be anodized. (a) n-type 6H-SiC wafer; (b) wet thermal oxidation; (c) LPCVD poly-Si; (d) poly-Si etching by RIE; (e) SiO_2 etching by HF solution; and (f) Ni sputtering and annealing.

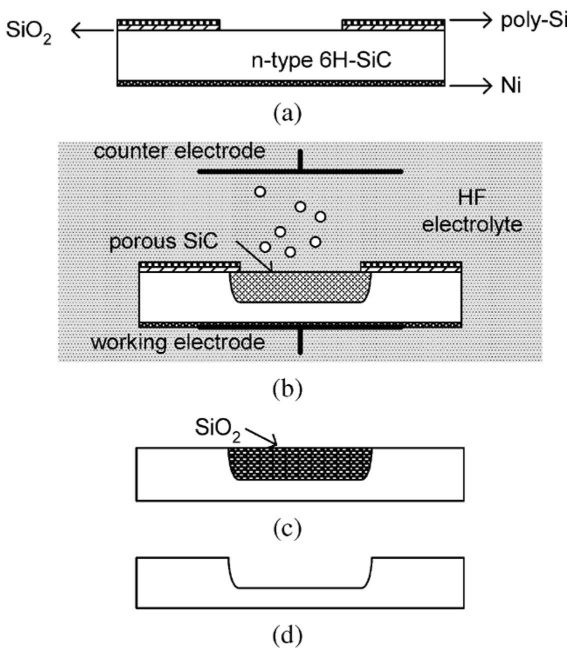

Fig. 2. Main process steps of the etching process for n-type 6H-SiC. (a) Sample with masking layers and back-side contacts; (b) anodization; (c) thermal oxidation; and (d) removing the oxide layer in concentrated HF.

which serves as working electrode, is attached to a Teflon holder and sealed with wax in order to protect the metallization on the backside of the sample. A platinum plate is used as counter electrode. The working and counter electrodes are connected to a constant current source. During the etching, the voltage and current are monitored by multimeters and recorded by a computer with GPIB interface. The anodization process is carried out at 20 °C.

III. Results and Discussion

A. Anodization Process

Cyclic voltammetry is used for studying the anodization process of the n-type 6H-SiC samples. The potential between anode and cathode is swept from 0 to 60 V with a step of 10 mV/s increasing and then decreasing. A typical current density versus voltage characteristic, the j–V curve, of the etching sample is illustrated in Fig. 3. Below the breakdown voltage, there is a leakage current of about 22.5 μA/cm^2. At about 2.5 V, the junction between n-type 6H-SiC and electrolyte starts to break down and the anodization of the 6H-SiC commences. After the breakdown, the current density rises sharply. When the applied voltage becomes larger, the increase of the current density becomes lower. When the voltage is large enough, 48 V in our case, there is a peak, which may indicate a change in the etching mechanism. If the applied voltage is further increased, the correspondent current density increases sharply and a lot of bubbles are generated. Further rising of the voltage makes the corresponding current density unstable. The current density decreases gradually as the applied voltage decreases from 60 back to 0 V.

Prepared samples are anodized in a 2 wt.% HF with a constant current density of 50 mA/cm^2 for one hour, and the change of the voltage during the anodization process is recorded. As we can see from Fig. 4, the voltage first increases to about 65 V, then decreases and becomes stable at a voltage of about 30 V. It takes about 5 min to stabilize the voltage. After the anodization, the samples are cut with a dicing saw for examination. A typical cross-section is illustrated in Fig. 5. 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 distinguished. The anodized surface (area 1) has the finest pores with a diameter of 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) which extends to the substrate has pores with a diameter of about 1 μm and a pore wall thickness of about 100 nm. The thickness of the pore walls is an important factor for etch result. If the pore walls in area 3 are too thick or nonuniform, they cannot be fully oxidized (and consequently removed) because the thermal oxidation rate of SiC is quite low in comparison with silicon. The etching parameters electrolyte concentration and current density have to be optimized in order to obtain a uniform pore size and hence, a smooth etched surface. It is determined experimentally that the use of a 2 wt.% HF electrolyte results in a pore structure, whose uniformity and thickness are superior to that obtained with a higher concentration of HF. At the same time, the use of a 2 wt.% HF solution does not require high voltages, as is the case for lower HF concentration because of their low conductivity. Fig. 6 illustrates a sample etched with an 8 wt.% HF electrolyte. The anodized porous area exhibits a nonuniform porous structure and irregular profiles in lateral and

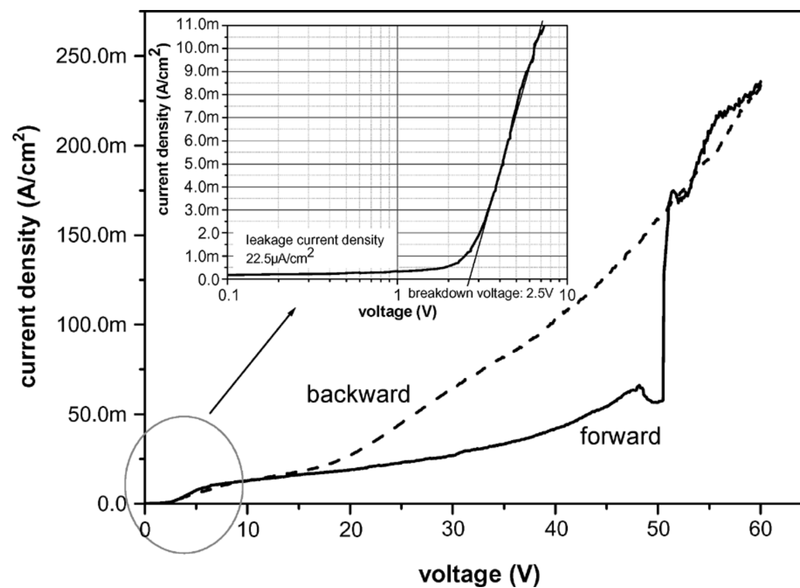

Fig. 3. Typical j–V curve of n-type 6H-SiC in 2 wt.% HF.

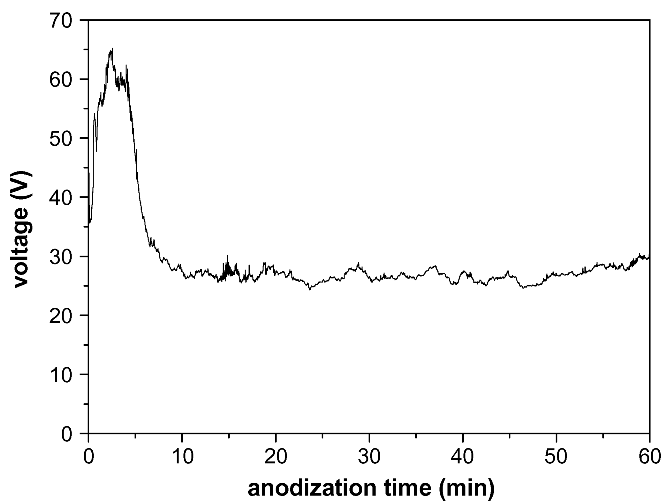

Fig. 4. Typical V-t curve of n-type 6H-SiC in 2 wt.% HF at 50 mA/cm^2.

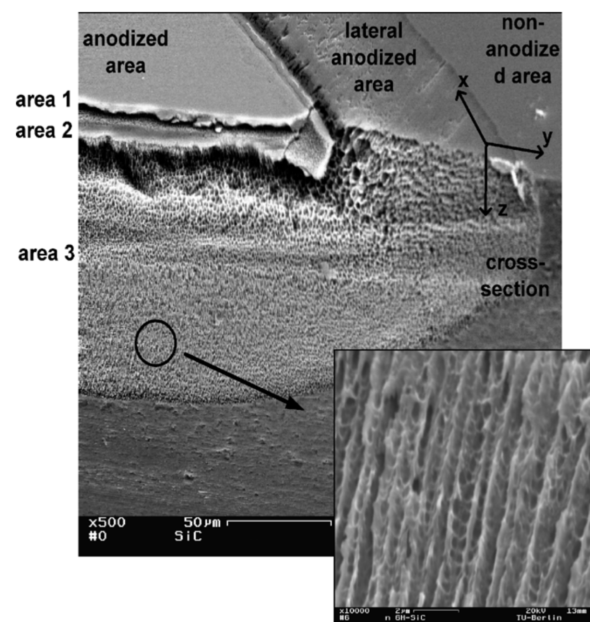

Fig. 5. Cross-section of an n-type 6H-SiC sample after 2 h anodization (50 mA/cm^2, 2 wt.% HF).

vertical direction. Such results make the thermal oxidation difficult and the etching profile irregular. Similar results are found for the current density that is used to anodize the SiC samples. With a high current density (200, 300 mA/cm^2), a violent reaction will occur and generate a large number of bubbles, which results in a poor porous structure and irregular etching profile. A lower current density yields a more uniform porous structure but it reduces the anodization rate. As a result, a current density of 50 mA/cm^2 is recommended as the optimum value for the process. The corresponding anodization rate is determined to be about 1.1μm/min.

B. Oxidation Process

After the anodization process, the samples are oxidized in a wet thermal oxidation environment at a temperature of 1150 °C for 10 h. Fig. 7 shows the cross-section of an oxidized 6H-SiC porous layer. The pore walls grow thicker upon oxidation and thus make the pores smaller and thinner.

The oxidized samples are then immersed in a concentrated HF solution to remove the thick porous oxide layer. After this step, the porous structure can still be seen at the bottom of the cavities, as shown in Fig. 8. The remaining pore tips have thick walls and could not be fully removed, even after a 24 h oxidation step. However, the depth of the remaining pores is less than 1 μm. The etched depth with respect to the anodization time is illustrated in Fig. 9. The relation shows a linear dependence, which means one can obtain the desired etching depth simply by adjusting the anodization time.

Fig. 10 illustrates the profile of an n-type 6H-SiC circular cavity measured by a Tencor alpha-step profile meter. An etching depth of about 150 μm is achieved by time-controlled anodization. The bottom of the etched cavity shows some

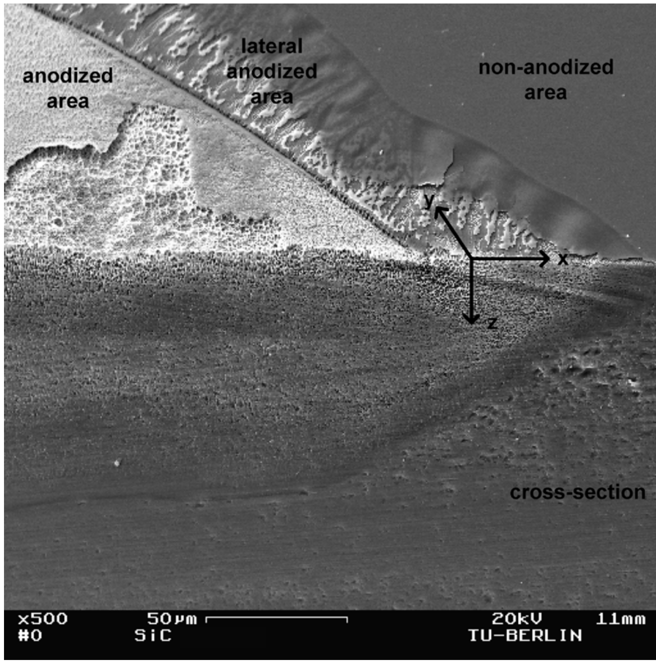

Fig. 6. Cross-section of an n-type 6H-SiC sample with a higher HF concentration (8 wt.%).

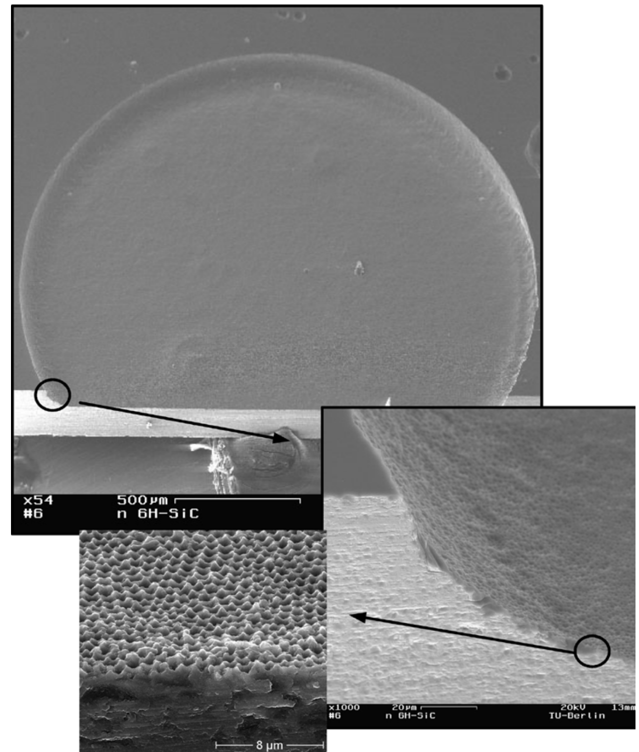

Fig. 8. Appearance of the porous structure after 10 h oxidation of 1150 °C. The oxidized porous layer is then etched in an HF solution.

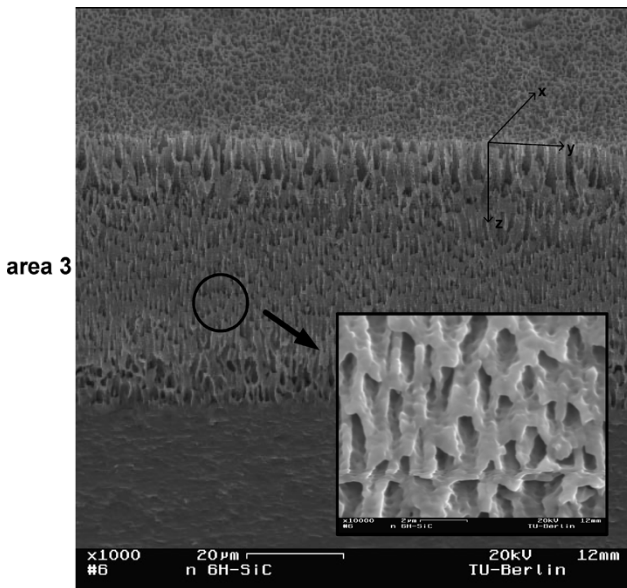

Fig. 7. Cross-section of the anodized n-type 6H-SiC sample after 10 h wet thermal oxidation after anodization in 2 wt.% HF.

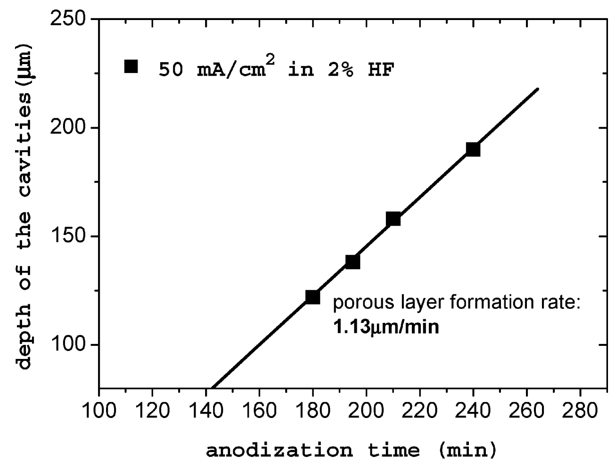

Fig. 9. Depth of the cavities versus anodization time.

uniformities. 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.

IV. Conclusion

A feasible method for the bulk micromachining of n-type 6H-SiC is presented in this paper. A suitable masking scheme and an ohmic contact metallization for the sample preparation are presented. It is demonstrated that it is possible to anodize n-type 6H-SiC substrates in an HF electrolyte solution without UV-light illumination. The proposed two-step process for deep 6H-SiC etching is investigated in detail. During the anodization process, a deep porous SiC layer is formed. The optimum anodization parameters for uniform pores and thin pore walls are found to be 2 wt% HF-electrolyte solution and a constant current density of 50 mA/cm^2. In the second process step, the porous layer is oxidized at 1150 °C for 10 h. The as-formed thick porous oxide layer is then removed in concentrated HF solution and the etched structure appears. Deep circular cavities are obtained by this method, which could be used for the formation of membranes for future SiC pressure sensors.

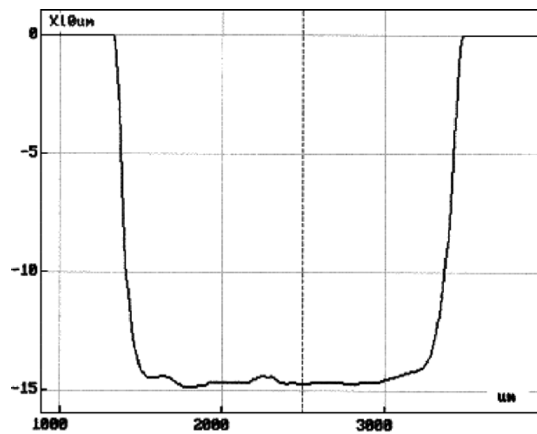

Fig. 10. Profile of the etched 6H-SiC cavity. The etching depth is about 150 μm.

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